# ROLE OF CEMENT AND RICE - HUSK ASH AS ADDITIVES IN STABILIZATION OF EXPANSIVE SOILS

*by*
PRADEEP KUMAR

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DEPARTMENT OF CIVIL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

DECEMBER, 1986

# ROLE OF CEMENT AND RICE - HUSK ASH AS ADDITIVES IN STABILIZATION OF EXPANSIVE SOILS

A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of

**MASTER OF TECHNOLOGY**

*by*
PRADEEP KUMAR

to the

**DEPARTMENT OF CIVIL ENGINEERING**

INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

**DECEMBER, 1986**

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# CERTIFICATE

Certified that the work on 'ROLE OF CEMENT AND RICE-HUSK ASH AS ADDITIVES IN STABILIZATION OF EXPANSIVE SOILS' by Sri Pradeep Kumar has been carried out under my supervision and this work has not been submitted elsewhere for a degree.

15th December, 1986.

(K.V.G.K. GOKHALE)
Professor
Department of Civil Engineering
Indian Institute of Technology
Kanpur.

## ACKNOWLEDGEMENTS

The present work is an outcome of the constant guidance, constructive suggestions and continuous help received from Dr. K.V.G.K. Gokhale, Professor in the Department of Civil Engineering at I.I.T. Kanpur. The author is indebted to him for all the interest taken by him in this work at several stages and for going through the manuscript.

The author is thankful to Dr. R.P. Singh, Dr. B.C. Raymahashay and Dr. A.S.R. Sai for their constant encouragement and to Dr. T.R. Ramachandran, Professor of Metallurgical Engineering for his help in the interpretation of the scanning electron micrographs.

Sri K.V. Lakshmidhar, Tefhnical Officer in the Geotechnical Engineering Laboratory, has rendered a lot of help in the laboratory investigations and author deeply appreciates the same. The assistance given by Mrs. L. Vijailaxmi of Environmental Engineering is also highly appreciated.

The author records his deep sense of appreciation for the spontaneous assistance received from Sri Umashankar of the X-ray laboratory and from Sri V.P. Sharma of the Electron Microscope Laboratory. Assistance received from Sri M.L. Srivastava, Sri S.D. Dubey and Sri R.S. Dube is appreciated.

The author also wishes to record his thanks to his friends Arun Kumar, Arun Singhal, Dhruva, Daljeet, Kamlesh, Manoj, Rakesh, Ranjeet, Sanjeev, Sohail Ahmad, Vijai Pratap Singh and Sri S.P. Sharma for their encouragement. The neat typing of the thesis by Sri R.N. Srivastava needs special mention.

- Pradeep Kumar

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# ABSTRACT

Among the additives used for stabilization of expansive soils, lime and cement are the most popular ones. While enough literature is available on soil-lime stabilization, information on the cement stabilization is very scanty. In addition, details on the nature of reaction products developed during stabilization and mechanism of their growth are not reported in detail. Rice-husk ash, a potential source of silica, can be used as an additive in conjunction with cement. No published information is available on the development of strength and growth of cementitious products in a soil-cement-rice-husk ash system.

In the present study, stabilization of two expansive soils with cement, rice-husk ash and cemos as additives has been studied in terms of strength development and mineralogical changes occurring during different periods of ageing. Hydrated silicates and aluminates of calcium are the products of cement hydration in addition to calcium hydroxide resulting in an increase in the pH of the system. The latter participates in the reaction along with the other starting materials in the system as confirmed from X-ray diffraction data. While in the dry state, the Banda and Jhansi soils have exhibited unconfined strength values around 0.600 $Kg/cm^2$ and 0.725 $Kg/cm^2$ respectively, stabilization with cement alone has enhanced the respective values of strength to 15 $Kg/cm^2$ and 9 $Kg/cm^2$ on 28 days of ageing. Addition of rice-husk ash to a soil-cement system results in the formation of the

cementitious products in increasing amounts. Tobermorite developed after 28 days in case of soil-cement system, has been evidenced even after 7 days in samples of soil-cement-rice-husk ash system. Xonotlite and β-dicalcium silicates are also two new products formed due to the addition of rice-husk ash with soil and cement. Improvement of strength upto 19 $Kg/cm^2$ and 11 $Kg/cm^2$ for Banda and Jhansi soils on ageing could be achieved during their stabilization with cement and rice-husk ash. It has been observed that the soil-cement-rice-husk ash reactions can be accelerated by adding cemos (a stabilizer) as has been evidenced in X-ray diffraction traces and electron micrographs from the improved formation of the products and also reflected in theimprovement of unconfined strength of the systems upto about 23 $Kg/cm^2$ and 14 $Kg/cm^2$ respectively. In addition to the products formed as in the case of soil-cement-rice-husk ash, gyrolite is a new product developed in presence of cemos in the system.

Scanning electron microscopy has enabled an understanding of the fabric and material development in the soil-additive reactions. Fabric changes are distinct at different times of ageing. While flocculation ensues immediately in the system, the fabric modifies to a honeycomb type on continued reaction with lime. In addition, morphological characteristics for the individual products and their pattern of growth has also been clearly observed in the scanning micrographs. Ettringite, a product of soil and cement in short-term reactions, exhibits a typical needle shape particles while tobermorite has distinct tubular morphology. The calcium aluminate hydrate phases exhibit characteristic hexagonal outlines.

Symbols used in Figures 5.1 to 5.6

| | | |
|---|---|---|
| $T_1$ | = | Monocalcium aluminate hydrate |
| $T_2$ | = | Dicalcium aluminate hydrate |
| $T_3$ | = | Tricalcium aluminate hydrate |
| $T_4$ | = | Tetracalcium aluminate hydrate |
| $T'_2$ | = | Dicalcium aluminate silicate hydrate |
| $T''_4$ | = | Tetracalcium carbonate hydrate |
| $C_1$ | = | Calcium silicate hydrate (C-S-H I and C-S-H II) |
| $C_2$ | = | Dicalcium silicate hydrate |
| $C_4$ | = | Gyrolite |
| $C_5$ | = | Tobermorite |
| $C_6$ | = | Xonotlite |
| I | = | Illite |
| M | = | Montmorillonite |
| K | = | Kaolinite |
| Cal | = | Calcite |
| Q | = | Quartz |
| F | = | Feldspar |

# CHAPTER 1

## INTRODUCTION AND SCOPE OF STUDY

Soil-cement stabilization has been in practice for more than half a century. It is found that this method finds vital use in the construction of pavements and air-fields in nearly 40 countries. In India, such stabilization has been practiced in several places like in Gorakhpur (U.P.) as a base course in road construction or at Kusmi airport near Gorakhpur for stabilizing a sandy loam type of soil. In South India also, soil-cement has been widely accepted as a base course materials. The foundation of Chidambaram-Bhuwanagiri road and construction of Ramanathapuram-Mandapam road are some of the examples. Soil-cement stabilization has extensive scope in the border road construction in the Himalayan terrain.

The properties and structure of soil change with the addition of pozzolana cement. It is commonly known that soils with similar physical properties but of varying chemical composition vary widely in their strength characteristics on stabilization with time. Thus an interaction of the cement with the soil constituents plays an important role in the strength behaviour of soils. This is true practically for all types of soils ranging from extremely granular to dominantly cohesive ones.

It is not easy to determine the nature of the soil-cement interactions. Specialized equipment is required besides the conventional items available for routine testing of soils in a regular Soil Mechanics Laboratory. An inter-disciplinary approach is also essential for the detailed investigations. The changes in stabilized soil samples over the ageing periods can best be tracked using techniques like the X-ray diffraction and electron microscopy. A better understanding of the soil-cement interactions would bring in proper and effective procedures for the strength improvement of soils.

As the strength development in soil-cement stabilization is influenced by cement-soil-clay interactions resulting in the formation of various cementitious products, enough scope exists for the use of cement in conjunction with other additives that serve as a source of silica. In rice-producing countries like India, Bangladesh, Sri Lanka, Burma or Thailand, rice-husk (an agricultural waste material) is available in enormous quantities. This material is used commonly as a fuel in the rice mill boilers and the ash that is produced on its burning is available source of silica. Rice-husk ash contains semi-amorphous silica upto around 85 to 90 percent which is highly chemically reactive. Thus if used in conjunction with cement, this material aids in the generation of silica-rich cementitious compounds.

Although enough work on strength characteristics has been reported on soil-cement stabilization, very little

is known about the soil-cement interactions and the mode of formation of compounds responsible in the strength development as is evident from the review of literature presented in the next chapter. In addition, no work exists on the use of rice-husk ash in soil-cement stabilization. In the present work an attempt has been made to understand (a) the mineralogical changes during soil-cement stabilization of typical expansive soils containing montmorillonite and illite, (b) the type of cementitious products and their morphology, (c) an understanding of the strength behaviour in soil-cement stabilized soils in terms of the soil-additives interactions, (d) the role of rice-husk ash in soil-cement stabilization for the improvement of the strength behaviour and (e) effect of cemos on soil-cement-rice-husk ash system in the fields of strength and formation of cementitious products.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 GENERAL

Treatment of expansive soils with chemical additives for the improvement of their engineering properties suitable for construction purposes has been in practice for several years. Of the different materials available for use as additives, cement has been very popular and widely adopted. Enough literature exists on soil-cement stabilization covering various aspects. In 1935, an extensive programme of research was initiated on soil-cement by the Portland Cement Association in the U.S.A. and the first experimental road was also laid near Johnsonvilla in the same year. During World War II, cement stabilization has been extensively practiced for construction and between 1941 and 1944, around 18 million square metres of airfield pavement were constructed with soil-cement as a subgrade. Since the start of the first soil-cement paving in 1935, the main objective has been to produce a dependable and long-life material. Efforts have been focused on several aspects such as selection of the cement content, moisture level and density on one hand and strength development on the other. Role of various chemicals in the development of strength has also been investigated in the last three decades. Procedures have also been evolved for an effective

way of mixing and compacting of soil-cement mixture during construction at the site. The literature reviewed here pertains to the strength characteristics and the nature of soil-cement interactions.

## 2.2 STUDIES ON THE STRENGTH CHARACTERISTICS

Addition of pozzolana cement to the soil changes the properties and structure of the soil. The unconfined compressive strength of the soil-cement increases with time of curing as in the case of concrete. Circeo, Davidson and David (1962) have established logarithmic and semi-logarithmic relations for the increase of compressive strength with the time of curing on the basis of statistical analysis of considerable data on granular, silty and clayey soil-cement samples. The relationship obtained by them has been used to predict the compressive strength of soil-cement for any particular mix cured for a specific period of time. According to these authors, the slope of the strength-age plots can be used as an indicator of the quality of a soil-cement mixture.

Several methods for determination of the cement content of soil-cement mixture have been reported by Curtis and Forbes (1963). These methods enable a rapid and accurate determination of cement content for use in construction techniques and design considerations. The time required for a complete determination varies from 1 to 8½ hours. Rude (1965) proposed a rapid method for

the determination of cement content of cement-treated base materials. Cement content is determined from a standard curve on the basis of calcium content present after subtracting the calcium in the aggregate. Davidson and coworkers (1962) have established that the optimum moisture content for maximum density and optimum moisture content for maximum unconfined compressive strength of cement-treated sand-clay mixtures are not necessarily the same. The moisture contents for maximum strength have been indicated to be on the dry side for sand-clay mixtures with sand as the dominant constituent while they are on the wet side for mixtures dominant in clay. Davidson and coworkers postulated that the variations between optimum moisture content for maximum density and that for the maximum strength are related to the particle size in the soil. According to these authors, soils having particles with larger surface area absorbed much of the added water so that insufficient water is available for the hydration of cement. Lightsey et al. (1970) have concluded on the basis of a detailed study that 2 to 4 percent of excess compaction moisture significantly improves the strength and durability depending on the type of soil and the delay time between mixing and compaction.

The elastic behaviour of soil-cement mixes has been studied by Reinhold (1955). The stress-strain curve in the case of a soil-cement mix has been observed to be concave with respective to the strain axis. The plastic

deformation in the first one-third of the failure load was reported to be extremely small and the material was considered as perfectly elastic within this range. With the stress increasing to 0.6 of the failure load, the stress-strain plot is reported to exhibit an increase in the curvature. It has also been observed that the clay content in the soil also increases the elasticity of the soil-cement mixes and that optimum quantity of clay to produce the maximum elasticity is about 25 percent. The various engineering properties of soil-cement as a construction material for road and airfield pavements have been summarized by Bhatia (1967). The typical terms of equipment used in different types of mixing have also been detailed by him.

## 2.3 EFFECT OF CHEMICAL ADDITIVES ON THE STRENGTH CHARACTERISTICS

Extensive work has been carried out by several workers on improving the properties of soil-cement with the use of chemical additives. The investigations centered on two primary objectives:

(1) to increase the effectiveness of portland cement as a soil stabilizer so as to reduce the quantity of cement required to treat expansive soils and

(2) to identify the exact trace chemicals that will enhance the effectiveness of cement as a stabilizer for soils that cannot be stabilized economically with cement alone.

Lambe and coworkers (1957, 1959) have established that sodium hydroxide is effective in improving the strength of all soils having low to moderate amounts of organic matter. The effectiveness of a sodium compound decreases with increasing plasticity and organic matter in the soils. Uppal and Kapur (1958) have reported that addition of sodium chloride has not resulted in any improvement of the engineering properties of sandy and clayey soils. Moh and Michaels (1962) have observed that the cement stabilized clays are susceptible to sulphate attack after long periods of contact with sulphate solutions. They have also reported that the effectiveness of sodium hydroxide in clay-cement can be materially improved by treating heavy clays with secondary additives. The resistance of soil-cement exposed to sulphate has also been studied by Cordon (1962). According to him, the deterioration in soil-cement subjected to attack by sulphate salts is more rapid in the soil-cement mixtures than in the concrete primarily because the soil-cement is less dense and the sulphate solution penetrates at a rapid rate. However, it was also established that a small amount of sulphate salts mixed with soil-cement increases the compressive strength and the resistance of the material to the action of freezing and thawing. Coleman et al. (1962) have examined the use of fly ash and sodium carbonate as additives to soil-cement combinations. It has been reported that the addition of fly ash tends to retard the setting time of soil-cement mixtures, thus

allowing more time for mixing and accelerates the hardening of soil-cement-fly-ash mixtures. Laguros and Davidson (1963) have reported that the type and exact quantity of a chemical that is most beneficial for the soil-cement stabilization are unique for each soil and depend on the texture of the texture of the soil, the type of clay minerals and particularly on the acidity of the soil. The effects of addition of lime, sodium hydroxide and sodium carbonate in soil-cement stabilization have been investigated by these authors and the changes in the unconfined compressive strength in the soil-cement samples have also been presented. It has been reported in the literature (Catton and Felt, 1943; Clare and Sherwood, 1954 and Sherwood, 1962) that addition of 0.2 to 0.5 percent of calcium chloride helps in the stabilization of organic soils with cement. A drastic increase in strength was reported in silty loam type of soils with addition of 2.5 percent calcium chloride and upto 10 percent cement (Uppal and Kapur, 1958). The soils that generally give best results with calcium chloride belong to the 'B' horizon in a soil profile (Bhatia, 1967).

The durability of cement-stabilized soil under alternating freezing and thawing conditions has also been investigated. George and Davidson (1963) have designed a freeze-thaw test to simulate winter field conditions. The design criteria of the various strength ranges for cold climates have also been detailed. Packard and Chapman (1963) have reported several methods for measuring

deterioration of soil-cement in freeze-thaw and wet-dry tests. A comparative evaluation of this method has also been presented.

## 2.4 SOIL-CEMENT INTERACTIONS

Herzog and Mitchell (1963) have studied the nature of reactions of clay with portland cement. Their investigations included stabilization of kaolinite and monomorillonite with cement as also with tricalcium silicate. According to them, clay-cement cannot be regarded as a simple mixture of hydrated cement particles holding together unaltered clay particles, but should be considered as a system in which both the clay and the hydrating cement combine through secondary reactions. However their study was only with pure clay minerals and not soils. Moh (1965) studied the reactions of soil minerals with cement and indicated the mechanism of stabilization. The reaction scheme of a soil stabilizer system has been outlined by him. The influence of soil mineralogy on soil-cement stabilization and the structures of stabilized soils have been studied by Croft (1967a, 1967b).

The structural arrangement of the reaction products and the soil constituents have been observed to contribute to the rigidity of the stabilized soils. According to Croft, soils that are well stabilized with cementitious agents can be regarded as brittle materials with elastic behaviour.

## 2.5 RICE-HUSK ASH AS AN ADDITIVE IN SOIL STABILIZATION

Studies with rice-husk ash as a material for soil stabilization have been very limited. Such studies have been initiated at Bangkok by Lazaro and Moh (1970) who found that addition of lime and rice-husk ash to clayey soils of Bangkok reduced the plasticity and swelling characteristics and improved the strength properties. The strength gain in the early ageing period has been attributed by these authors to the soil-lime reactions while further gain in strength with longer ageing was attributed to the lime-rice-husk ash reaction also. Ramaiah et al. (1972) reported the engineering properties of expansive soils with addition of rice-husk ash. Jose James and Subba Rao (1986a), in a paper on the reactivity of rice-husk ash with clayey soils, correlated the reactivity with the calcium ion concentration. According to them, the reaction does not take place when the concentration of calcium ions in solution is below 2 mmoles even with excess silica being present. They also concluded that the time required for 90% of the reaction to be completed depends on the initial $CaO:SiO_2$ ratio.

Very little information is available on the X-ray diffraction and electron microscopic data of stabilization process. Gokhale and Swaminathan (1983) gave the details of changes occurring in the fabric in mineralogy during stabilization in expansive soils with lime and lime-rice-husk ash. They used the various techniques including X-ray diffraction, differential thermal analysis and electron

microscopy. James and Subba Rao (1986b) gave the reaction products of lime and silica from rice-husk ash. According to them, rice-husk ash (containing about ~95% silica) with known physical and chemical characteristics reacting with lime and water gives calcium silicate hydrate and C-S-H I. They repeated their experiments with lime-excess and lime-deficient mixtures. These authors concluded that the product C-S-H I accounts for the strength of lime-rice-husk ash content.

# CHAPTER 3

# MATERIALS AND METHODS

## 3.1 GENERAL

In this chapter, the materials chosen for the study and methods adopted in sample testing have been described. Materials used in the work namely the two types of soils, cement, rice-husk ash and cemos have been characterized in regard to their physical and mineralogical properties. The procedures adopted for sample preparation and also the techniques involved in the characterization of starting materials as well as the samples at various times of ageing during stabilization are outlined in detail.

## 3.2 MATERIALS USED

### 3.2.1 Soil

Black cotton soil samples from Jhansi and Banda have been chosen for study. The soil from Jhansi is grey in colour and is moderately plastic containing 50 percent of clay fraction (Figure 3.1). The physical and engineering properties of the soils are presented in Table 3.1. X-ray diffraction pattern (Figure 3.2) of soil sample has shown the presence of quartz along with montmorillonite, illite, feldspar, kaolinite and calcite. The 'd' spacing values for all these soil constituents are listed in Table 3.2.

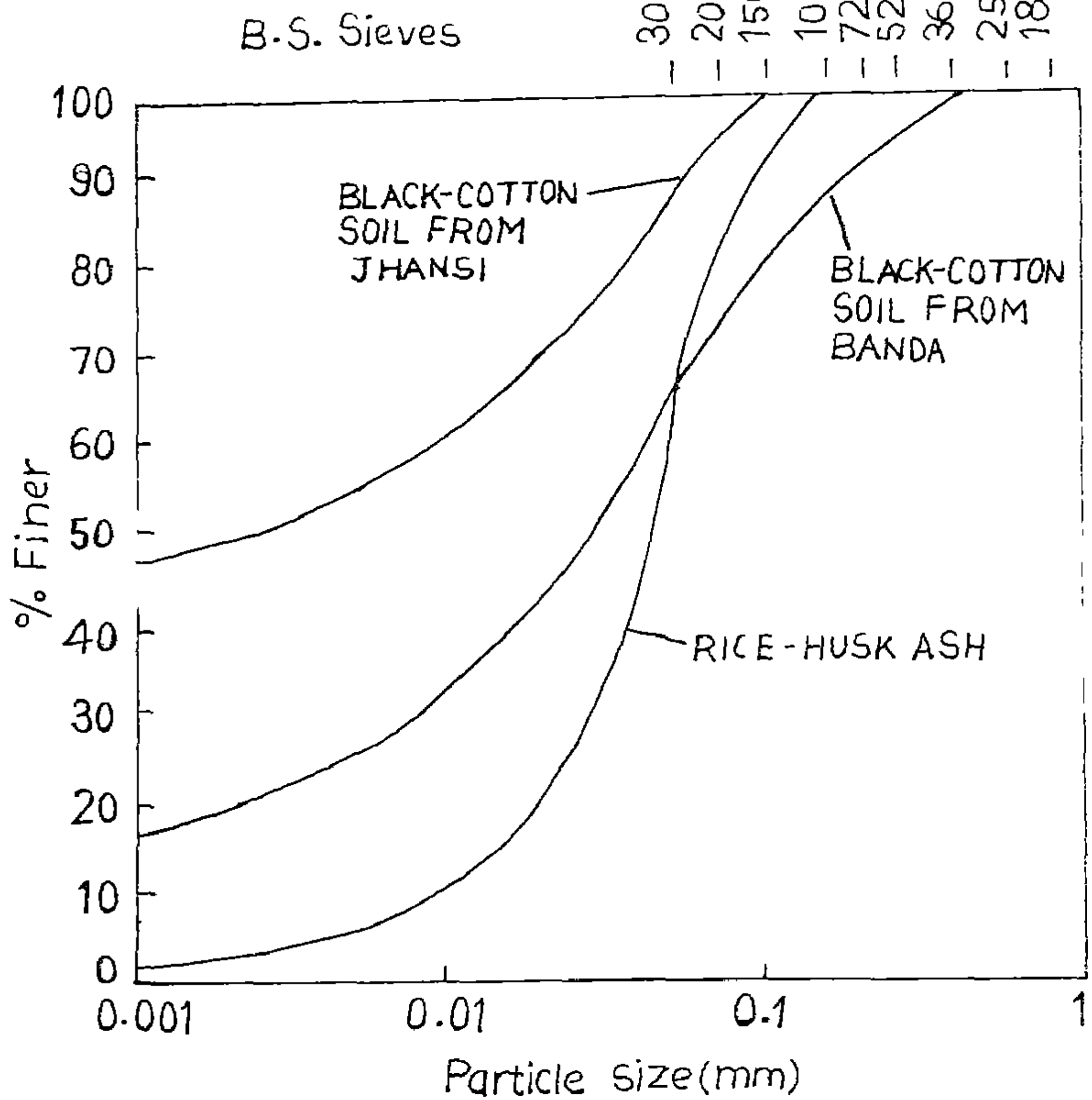


FIG-3.1: GRAIN SIZE DISTRIBUTION CURVES

TABLE 3.1 : SOIL PROPERTIES

| Properties | Soil from Banda | Soil from Jhansi |
|---|---|---|
| Liquid limit, percent | 40 | 48 |
| Plastic limit, percent | 18 | 24 |
| Plasticity index | 22 | 24 |
| Shrinkage limit, percent | 8.50 | 11.00 |
| Specific gravity, gm/cc | 2.55 | 2.65 |
| pH | 7.7 | 9.1 |
| Dry density, gm/cc | 1.95 | 1.85 |
| Optimum moisture content, percent | 21 | 25 |
| Unconfined compressive strength (with optimum moisture content), $Kg/cm^2$ | 0.600 | 0.725 |
| Clay (<0.002 mm), percent | 20 | 50 |
| Silt (0.06-0.002 mm), percent | 45 | 40 |
| Sand (2.0-0.06 mm), percent | 35 | 10 |

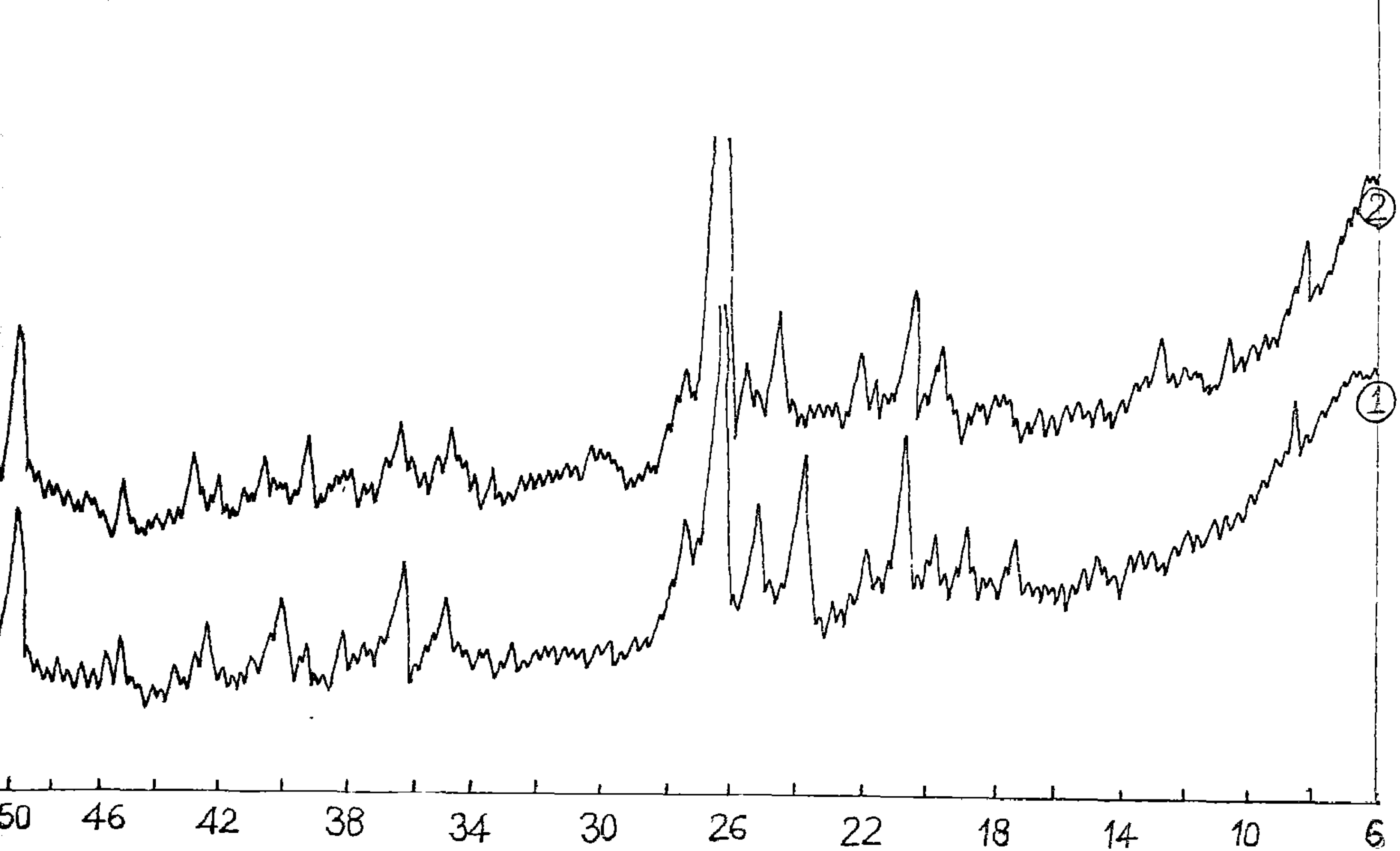


FIG.3.2: X-RAY DIFFRACTION PATTERNS OF BLACK COTTON SOIL
(1-BANDA, U.P. 2-JHANSI, U.P.)

TABLE 3.2 : X-RAY DIFFRACTION DATA FOR BLACK COTTON SOIL FROM JHANSI (U.P.)

(RADIATION : $CuK_{\alpha}$)

| $2\theta$ (degrees) | d (Å) | Constituents |
|---|---|---|
| 8.90 | 9.92 | Illite |
| 12.60 | 7.01 | Kaolinite |
| 19.80 | 4.48 | Montmorillonite |
| 20.90 | 4.24 | Quartz |
| 22.30 | 3.98 | Feldspar |
| 22.90 | 3.88 | Illite |
| 24.20 | 3.67 | Montmorillonite |
| 25.40 | 3.50 | Montmorillonite |
| 25.80 | 3.44 | Kaolinite |
| 26.60 | 3.35 | Quartz |
| 27.70 | 3.21 | Montmorillonite |
| 34.70 | 2.58 | Illite |
| 35.20 | 2.54 | Illite |
| 35.20 | 2.54 | Montmorillonite |
| 36.70 | 2.44 | Quartz |
| 37.90 | 2.37 | Kaolinite |
| 39.60 | 2.27 | Quartz |
| 40.40 | 2.23 | Quartz |
| 42.50 | 2.12 | Quartz |
| 43.20 | 2.09 | Calcite |
| 45.70 | 1.98 | Quartz |
| 50.20 | 1.82 | Quartz |

TABLE 3.3 : X-RAY DIFFRACTION DATA FOR BLACK COTTON SOIL FROM BANDA (U.P.)

(RADIATION : $CuK_{\alpha}$)

| 2θ (degrees) | d (Å) | Constituents |
|---|---|---|
| 8.90 | 9.93 | Illite |
| 17.70 | 5.00 | Illite |
| 19.50 | 4.55 | Montmorillonite |
| 19.80 | 4.48 | Montmorillonite |
| 20.80 | 4.26 | Quartz |
| 21.90 | 4.05 | Feldspar |
| 22.90 | 3.88 | Illite |
| 23.50 | 3.78 | Feldspar |
| 23.95 | 3.72 | Montmorillonite |
| 25.40 | 3.50 | Montmorillonite |
| 26.60 | 3.35 | Quartz |
| 28.00 | 3.18 | Feldspar |
| 34.60 | 2.59 | Illite |
| 35.00 | 2.56 | Montmorillonite |
| 36.50 | 2.46 | Quartz |
| 38.10 | 2.37 | Montmorillonite |
| 39.40 | 2.29 | Montmorillonite |
| 40.20 | 2.24 | Quartz |
| 42.40 | 2.13 | Quartz |
| 45.40 | 1.99 | Illite |
| 45.60 | 1.98 | Quartz |
| 50.20 | 1.82 | Quartz |

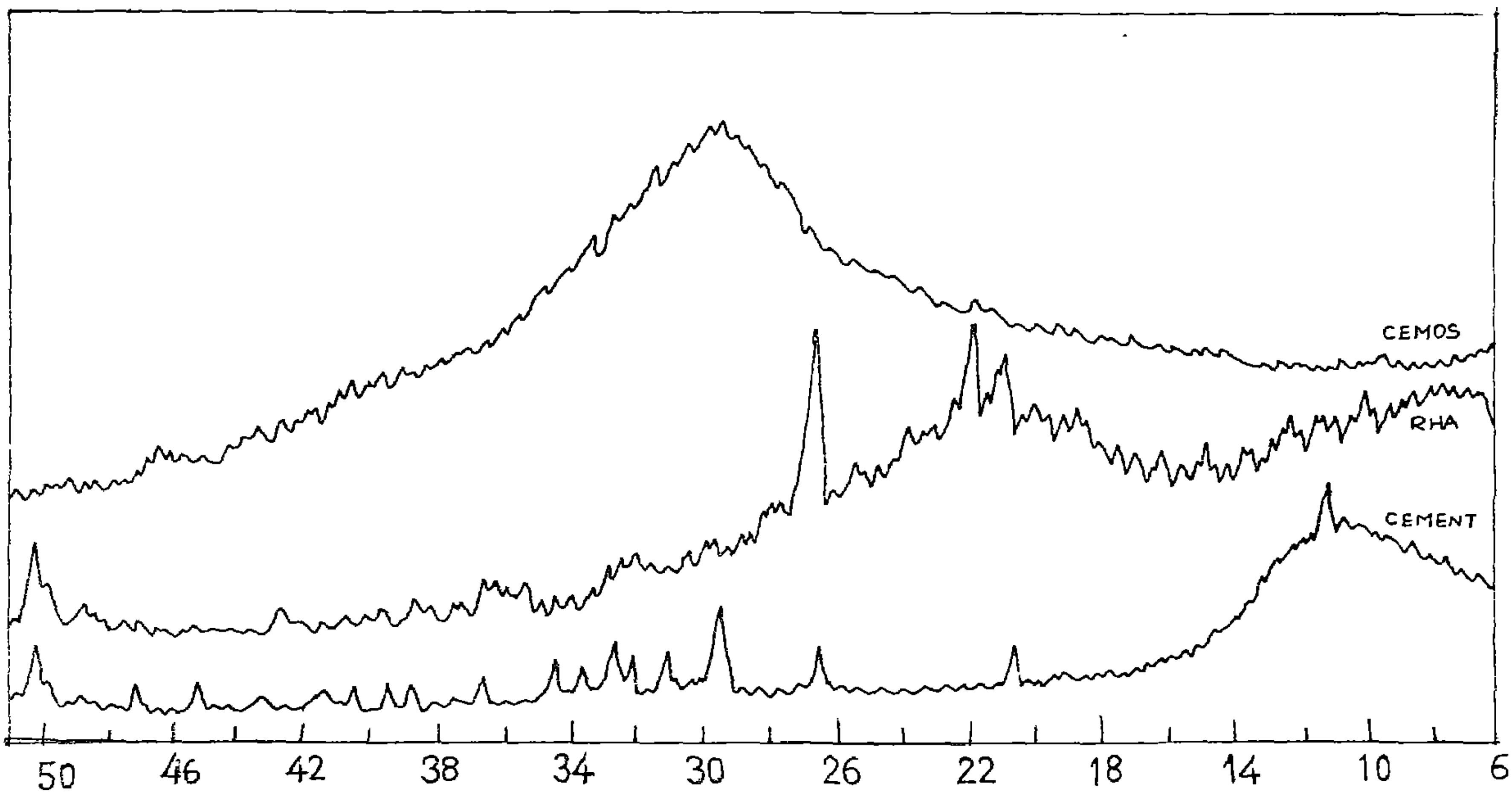


FIG.3.3: X-RAY DIFFRACTION PATTERNS OF ADDITIVES

TABLE 3.4 : X-RAY DIFFRACTION DATA FOR POZZOLANA CEMENT USED

(RADIATION : $CuK_{\alpha}$)

| 2θ (degrees) | d (Å) | Constituents |
|---|---|---|
| 11.70 | 7.56 | Gypsum |
| 20.80 | 4.27 | Quartz |
| 20.80 | 4.27 | Gypsum |
| 26.60 | 3.35 | Quartz |
| 29.10 | 3.06 | Gypsum |
| 29.40 | 3.04 | Tricalcium silicate |
| 31.20 | 2.86 | Dicalcium silicate |
| 31.20 | 2.86 | Gypsum |
| 32.20 | 2.77 | Tricalcium silicate and Dicalcium silicate |
| 32.60 | 2.74 | Dicalcium silicate |
| 33.10 | 2.70 | Tricalcium silicate |
| 33.80 | 2.65 | Tetracalcium aluminoferrite |
| 34.40 | 2.60 | Tricalcium silicate |
| 36.70 | 2.45 | Quartz |
| 39.00 | 2.30 | Tricalcium silicate |
| 39.40 | 2.28 | β-dicalcium silicate |
| 41.30 | 2.18 | Tricalcium silicate |
| 43.30 | 2.08 | Dicalcium ferrite |
| 45.80 | 1.98 | Quartz |
| 47.40 | 1.92 | γ-dicalcium silicate |
| 50.20 | 1.82 | Quartz |

The soil from Banda is also grey in colour and moderately plastic. It contains about 20 percent of clay fraction. The grain size characteristics of the soil are indicated in Figure 3.1. X-ray diffraction pattern for the soil has revealed the presence of quartz, montmorillonite, feldspar, illite as the constituents. The characteristic reflections for these minerals are shown in Table 3.3. Montmorillonite is the dominant clay mineral in both these soils. Its proportion in the Jhansi soil is relatively more than in the Banda soil. Presence of calcium carbonate is seen in the soil from Jhansi as evident from the characteristic X-ray diffraction patterns.

### 3.2.2 Cement

The cement used in the analysis was portland-pozzolana cement. The X-ray diffraction pattern (Figure 3.3) for this material has indicated the presence of tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite as the main constituents with small amount of quartz. The characteristic peaks for each of these compounds is indicated in Table 3.4. Presence of gypsum in minor proportion is also evidenced from the presence of reflections around ($2\theta$) values of 11.7°, 20.8° and 29.1° corresponding to the 'd' values of 7.56 Å, 4.27 Å and 3.06 Å.

### 3.2.3 Rice-Husk Ash

Rice-husk ash used in the present study was prepared from the husk obtained from a rice mill in Kanpur. The material is greyish black in colour with a specific gravity of 2.01. Grain size distribution curve for the same is indicated in Figure 3.1. X-ray diffraction analysis of rice-husk ash sample has revealed the presence of quartz (evidenced from the characteristic peaks around 4.22 Å and 3.33 Å.

### 3.2.4 Cemos

Cemos 110, used as a stabilizer, has been obtained in liquid form. The X-ray diffraction pattern has indicated absence of peaks signifying its amorphous nature. It is reported to have been commonly used in hydrophobic concrete besides its use as stabilizer. The pH value for this material is 9.0.

## 3.3 METHODS

### 3.3.1 Sample Preparation

For each of the two soils, compacted samples with the following combinations were prepared:

Soil with 5% cement

Soil with 5% cement and 15% rice-husk ash

Soil with 5% cement, 15% rice-husk ash and 2% cemos.

In addition to the above, compacted samples of the two soils were also prepared for obtaining the initial strength values. The samples were compacted to the maximum

dry density with optimum moisture content. Several specimens were prepared in each case (soil, soil-cement, soil-cement-rice-husk ash and soil-cement-rice-husk ash-cemos) with differing moisture content and with Proctor's compaction device, the optimum moisture content was determined for each of these combinations.

For strength test, the specimens of $1\frac{1}{2}$" diameter and 3" height were prepared using the Dietert's compaction device. The soil, initially pulverized to pass through I.S. sieve 170 and oven-dried was mixed well with appropriate proportion of the additive materials to obtain a uniform dry mixture. The mix in each sample was properly mixed and kneaded with water equivalent to optimum moisture content. Then the mix was placed in a cylindrical mould of Dietert's compaction device and compacted with 10 blows on either side of the mould using a hammer of 18 lbs. having a free fall of 2" height. The double plunger action resulted in achieving a comparatively uniform density over the entire specimen. The specimen was then extruded with the help of hydraulic jack. The density in each case was confirmed by measuring the specimen dimensions and its weight. Immediately on preparation, the specimens after being given proper identification marks, are wrapped in polythene bags and sealed with tape to prevent carbonation of cement as also any possible evaporation of the moisture during ageing to different periods of time. Twelve specimens in each case have been prepared for testing 3 of them after each ageing

period (24 hours, 7 days, 14 days, 28 days). In all, 78 samples have been prepared and tested.

3.3.2 Testing for Compressive Strength

For the unconfined compressive strength determination, a compressive testing unit fitted a 234 Kg. proving ring complete with a dial gauge and a deformation dial gauge (of 10 mm travel and 0.01 accuracy) was used with the rate of loading maintained at 0.127 mm/min. The proving ring reading was taken at intervals of 10 mm of the dial gauge reading. The load was estimated from the proving ring readings by multiplying the same with the proving ring constant in each case.

3.3.3 Determination of pH Values

The values of pH were measured separately for both the soils, cement, rice-husk ash, cemos and other combinations by precision pH meter of Phillips make (model PR 9405/90) with a glass electrode. Estimates were made on fresh samples. Samples have been prepared taking 20 gms of the mix in each case dispersed thoroughly in 100 ml of distilled water. The suspension was shaken every 10 minutes for about a minute and at the end of an hour, the pH values have been recorded in each case.

3.3.4 X-ray Diffraction Studies

X-ray diffraction analyses have been carried out on ISO-DEBYEFLEX 2002D diffractometer of RICH SEIFERT make using

nickel filtered copper-K alpha radiation. Scanning was carried out between 7° and 50° (2θ) with a scanning rate of 3° (2θ)/min.

### 3.3.5 Electron Microscopic Investigations

In the present study, scanning electron microscopy was used. Samples prepared were examined in a I.S.I.-60 scanning electron microscope at 30 K.V. in secondary electron mode using suitable tilts. The photographs were obtained on ILFORD 35 mm film.

Soil-additive samples at different ageing periods were examined. A small amount of the sample in each case was dispersed in acetone. After the heavy particles settle, a few drops of liquid from the upper portion of the suspension is taken on an aluminium stub and allowed to dry. Carbon deposition has been carried out on the particles placed on the stub to improve the conductivity and signal to noise ratio. These samples are then examined under the scanning electron microscope.

# CHAPTER 4

# STRENGTH BEHAVIOUR IN SOIL ADDITIVE SYSTEMS

## 4.1 GENERAL

In the present work, pozzolana cement, rice-husk ash and cemos have been chosen as additives for stabilizing the expansive soils from Banda and Jhansi areas. Determination of unconfined compressive strength has been carried out on compacted cylindrical soil specimens with cement on one hand and cement and rice-husk ash on the other. Tests were also carried out with cemos in the soil-cement-rice-husk ash systems. The procedure for sample preparation and testing have already been detailed in the previous chapter. With different proportions of these additives, strength determinations have been carried out for specimens using Banda and Jhansi soils aged to 7, 14 and 28 days. The trends of strength variations have been analyzed. These variations are in conformity with mineralogical changes observed in the X-ray diffraction patterns of the respective soil additive mixes.

## 4.2 SOIL-CEMENT SYSTEMS

### 4.2.1 With Black-Cotton Soil from Banda

The variation in the soaked unconfined compressive strength for compacted specimens of the Banda soil on ageing to different periods of time are indicated in

Figure 4.1a. The soil has a compressive strength 0.60 $Kg/cm^2$ under dry conditions. With cement in the soil, an increase in strength has been observed on ageing. At the end of 24 hours, the soil specimens with 5 percent of cement registered compressive strength values around 5.0 $Kg/cm^2$. Strength gain has been observed to be upto 13 $Kg/cm^2$ at the end of 14 days and beyond this period, the rate of increase has decreased resulting in the samples attaining a final value of 15 $Kg/cm^2$ on ageing to 28 days.

### 4.2.2 With Black Cotton Soil from Jhansi

The trend for increase in the strength in this case on ageing is similar to that in the soil-cement samples using Banda soil (Figure 4.1b). In this case also, the rate of increase has shown a decreasing trend beyond 14 days of ageing. At the end of 28 days of ageing, the strength value has been observed to be around 9 $Kg/cm^2$.

## 4.3 SOIL-CEMENT-RICE-HUSK ASH SYSTEM

### 4.3.1 Black Cotton Soil from Banda

Strength variation values for soil-cement-rice-husk ash combination for Banda soil are indicated in Figure 4.2a. A distinct and rapid strength development on ageing could be evidenced from samples with 5 percent cement and

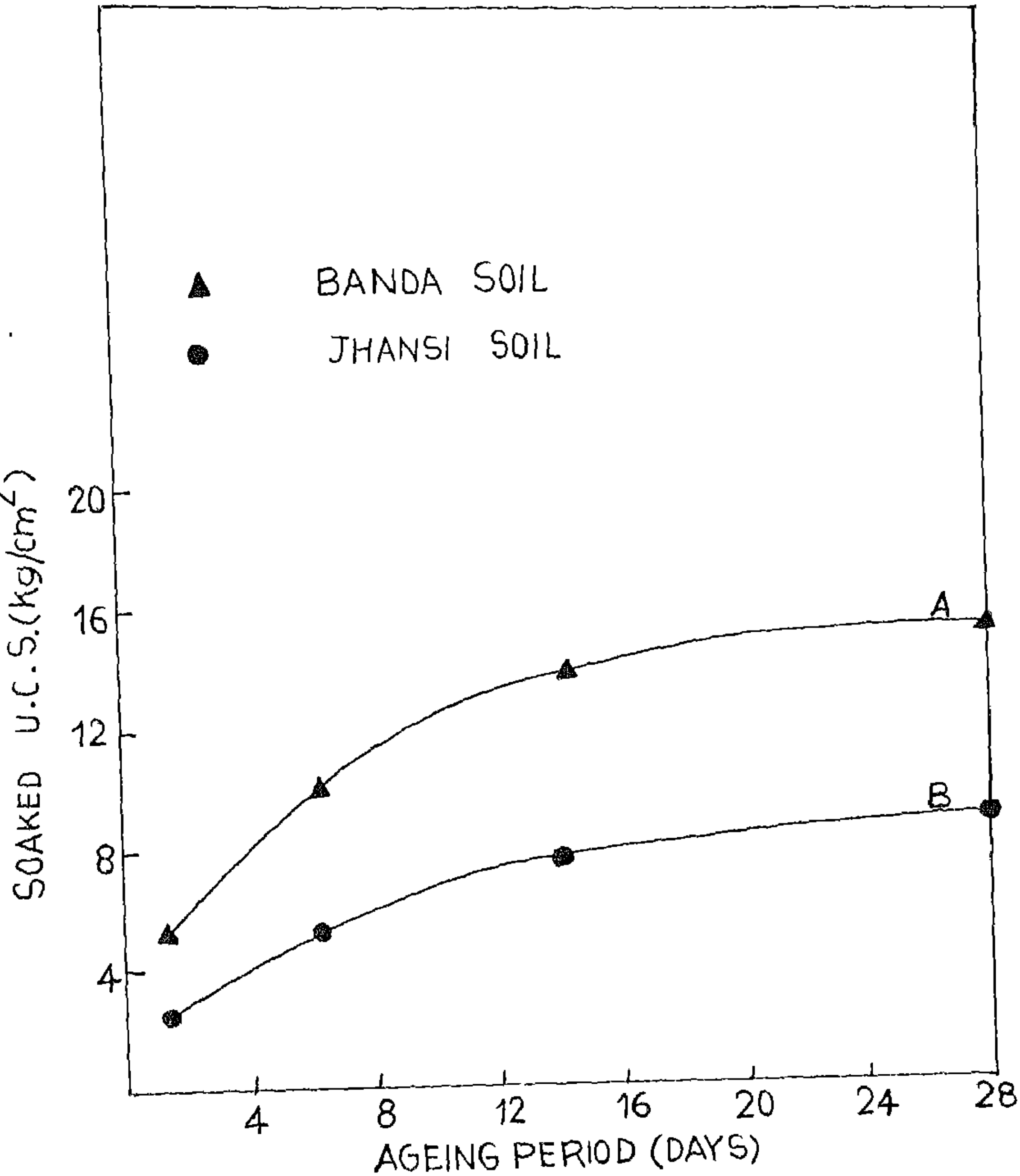


FIG. 4·1: STRENGTH VARIATIONS FOR SOIL WITH 5% CEMEN

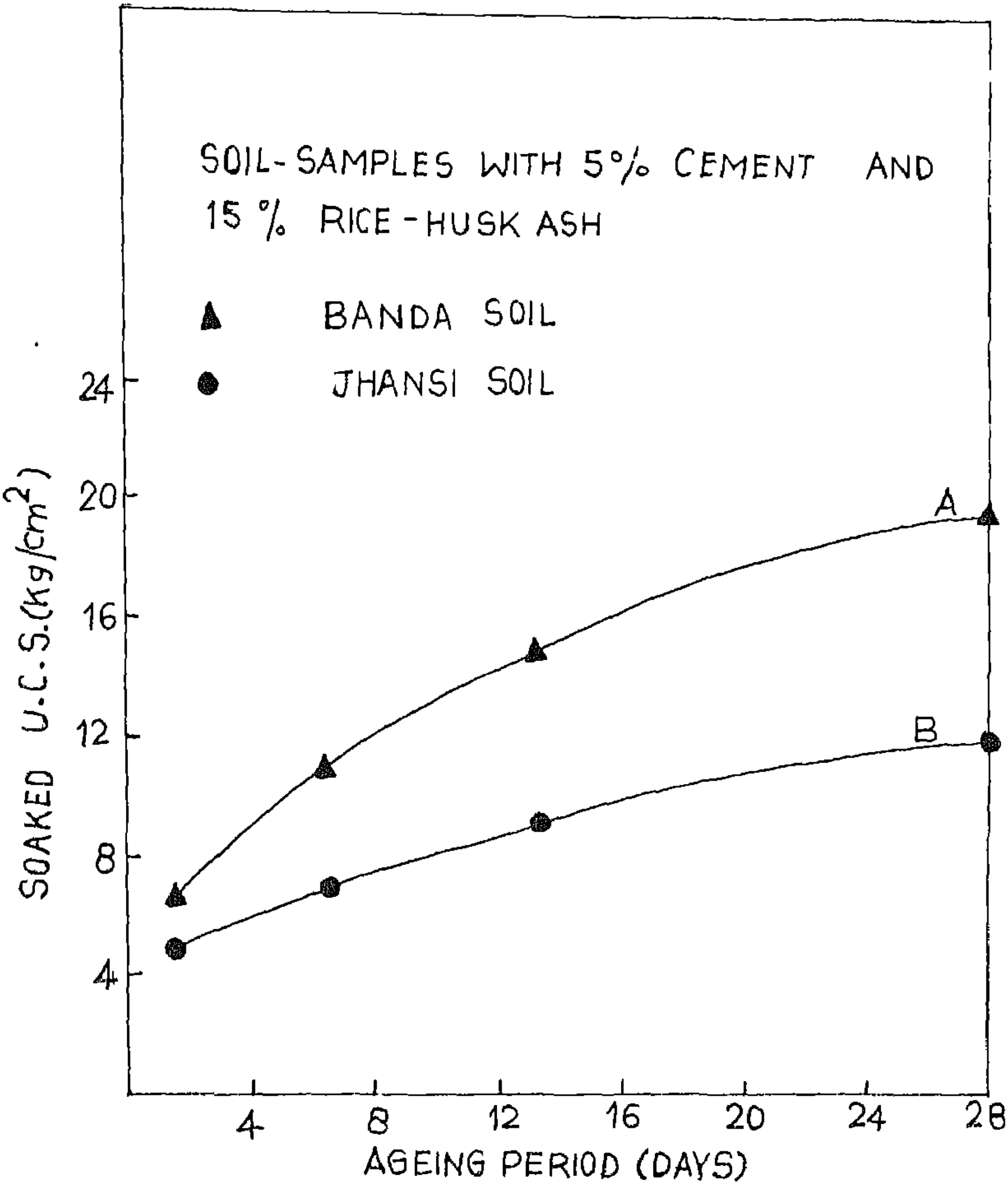


FIG.4.2: STRENGTH VARIATIONS FOR SOIL WITH CEMENT AND RICE-HUSK ASH

15 percent rice-husk ash in comparison to the samples with 5 percent cement only. Cement and rice-husk ash in 1:3 proportion have exhibit strength values around 6.6 $Kg/cm^2$ after 24 hours. Between 24 hours and 14 days, there is rapid increase in compressive strength from 6.6 $Kg/cm^2$ to 13.8 $Kg/cm^2$. After that upto 28 days of ageing, strength increase has registered a decreasing trend with the final value reaching around 19.2 $Kg/cm^2$.

4.3.2 Black Cotton Soil from Jhansi

Samples with cement and rice-husk ash combination have exhibited uniform strength gain between 24 hours and 14 days (Figure 4.2b). The compressive strength value at 24 hours is 4.8 $Kg/cm^2$. The rate of increase in strength in the samples on ageing is relatively at a slower pace compared to the corresponding samples using Banda soil. it may be indicated here that presence of rice-husk ash in the soil-cement system has resulted in considerable increase of strength upto 4.8 $Kg/cm^2$ at the end of 24 hours of ageing compared to the soil-cement sample, aged to same period, which has attained only strength values around 2 $Kg/cm^2$. However after 24 hours the strength development in soil-cement-rice-husk ash samples has been distinctly slowed down with the samples attaining values around 11 $Kg/cm^2$ on ageing to 28 days.

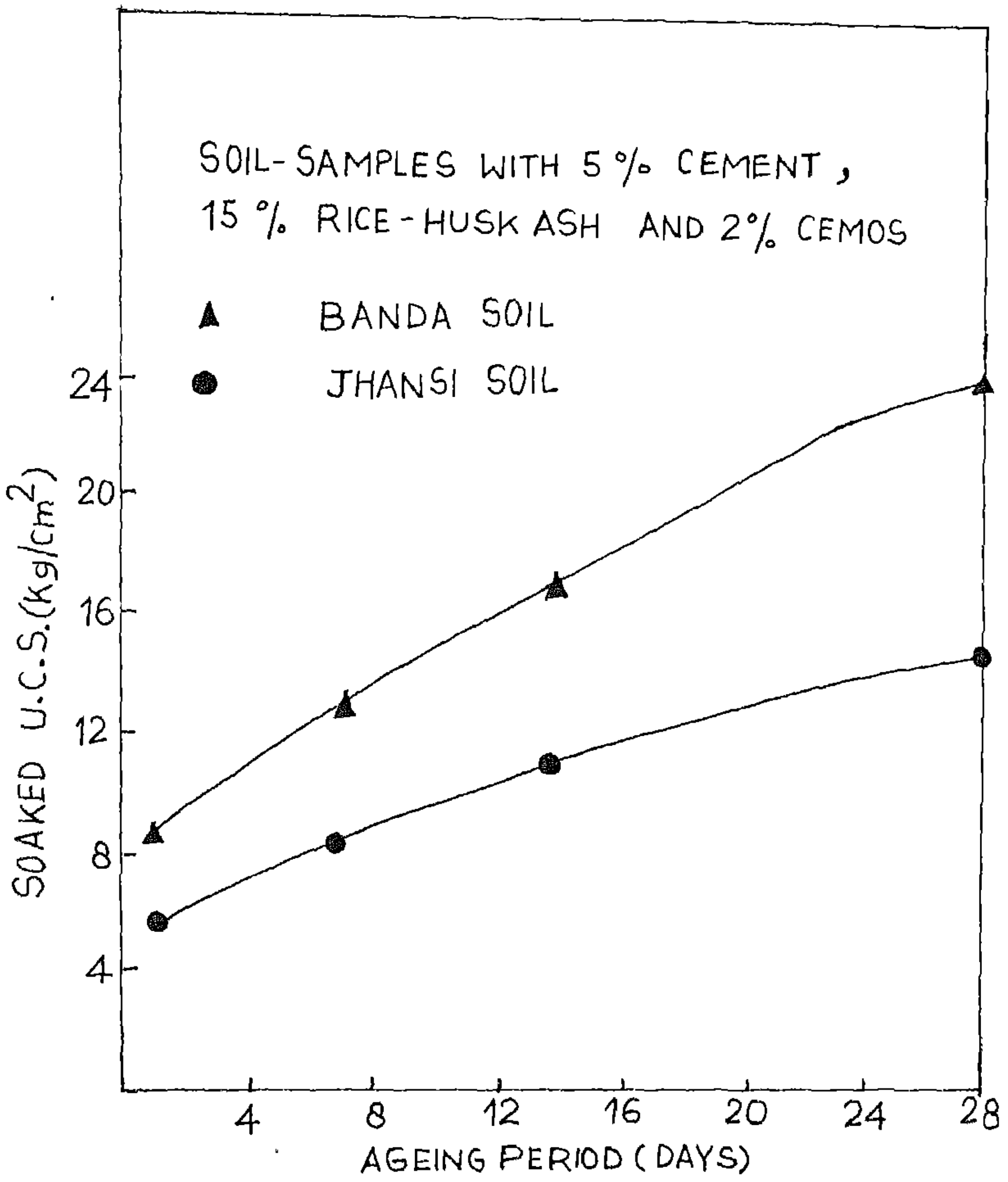


FIG.4·3: STRENGTH VARIATIONS FOR SOIL WITH CEMENT RICE-HUSK ASH AND CEMOS

## 4.4 SOIL-CEMENT-RICE-HUSK ASH-CEMOS SYSTEM

Presence of cemos in the soil-cement-rice-husk ash samples has affected the strength ageing to a considerable extent as evident from Figure 4.3. The strength gain has been observed to be at an uniform pace in the case of soil-cement-rice-husk ash system with Banda soil as compared to the corresponding sample with Jhansi soil where the rate of strength increment has been slowed down to some extent beyond 14 days of ageing. While in the case of Banda soil, the strength has increased from 8.5 $Kg/cm^2$ (at the end of 24 hours) to 23.2 $Kg/cm^2$ (at the end of 28 days), the increase has been from 5.5 $Kg/cm^2$ to 13.8 $Kg/cm^2$ for the corresponding period in the case of Jhansi soil. Thus the effect of cemos is more pronounced in the soil-cement-rice-husk ash system with Banda soil.

# CHAPTER 5

# X-RAY DIFFRACTION STUDIES

## 5.1 GENERAL

X-ray diffraction investigations form an important part of the study as they enable complete mineralogical characterization within the soil-additive system. Whereas a conventional chemical analysis would indicate an overall chemical composition in terms of constituents such as $SiO_2$ and $Al_2O_3$, the chemical data will not be indicative of the various individual mineralogical constituents present within the system at any particular time of ageing.

In the present study, samples of different soil-additive proportions (soil-cement, soil-cement-rice-husk ash and soil-cement-rice-husk ash-cemos) aged to differing periods of time have been systematically analyzed for black cotton soils from Banda and Jhansi using X-ray diffraction technique. In each case, the mineral constituents present within the system have been characterized to enable an understanding of the strength variations in the system on ageing.

## 5.2 CHANGES IN SOIL-CEMENT SYSTEM

### 5.2.1 Black-Cotton Soil from Banda

The X-ray diffraction patterns for samples treated with 5 percent cement are indicated in Figure 5.1. On ageing to 7 days, several changes can be noticed (Figure 5.1b).

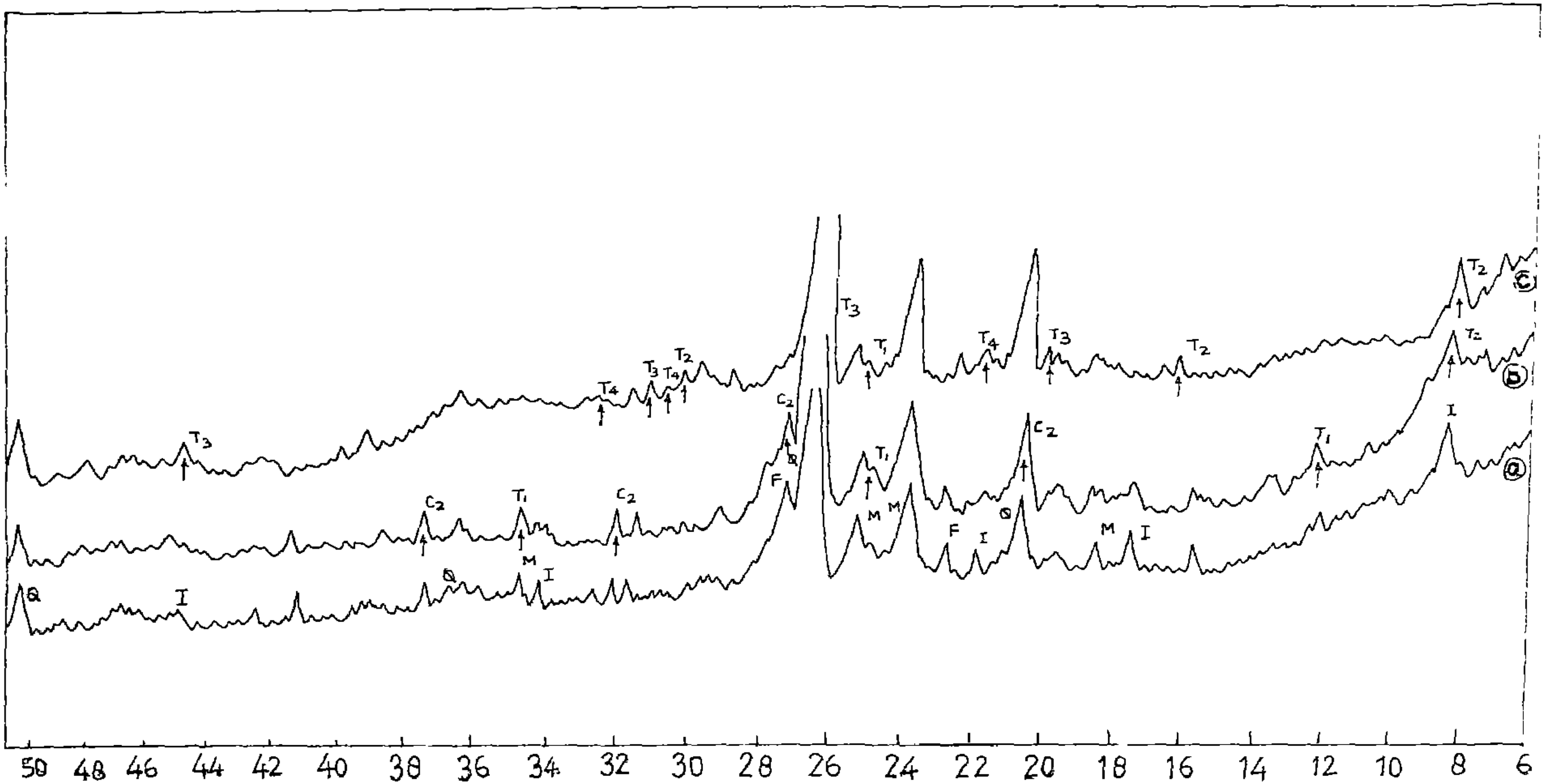


FIG. 5.1: X-RAY DIFFRACTION PATTERNS OF BANDA SOIL SAMPLES WITH 5 % CEMENT AGED TO (a) 24 HOURS (b) 7 DAYS (C) 28 DAYS

The formation of calcium hydroxide is distinctly seen from the appearance of its intense reflections around 4.90 Å, 2.63 Å, 1.93 Å and 1.79 Å. They start diminishing and totally disappear on ageing to 28 days (Figure 5.1c). This is indicative of the consumption of calcium hydroxide in reactions during ageing period. Reduction in the intensities of reflections for illite and montmorillonite has been clearly noticed in the X-ray traces. On ageing, the peaks for these minerals also appear relatively broader in addition to their reduced intensities. Thus it is evident that the clay minerals also participate in the reactions. An indication of the formation of new cementitious products in minor quantities is seen even at the end of 7 days of ageing from the appearance of small peaks around 5.61 Å, 4.69 Å, 3.88 Å, 2.77 Å, 2.56 Å and 2.21 Å. These reflections correspond to ettringite (calcium aluminate trisulphate hydrate). Reflections of monocalcium aluminate 10 hydrate can also be seen around 7.16 Å, 3.56 Å and 2.55 Å. In addition, the formation of α-dicalcium silicate hydrate has also been evidenced from the appearance of peaks around 4.22 Å, 3.27 Å, 2.80 Å and 2.41 Å at the end of 7 days.

On ageing to 28 days, several distinct changes have been observed in the system (Figure 5.1c). Ettringite which was present earlier appears to have been modified into a stable phase as evidenced from the absence of its characteristic peaks. In addition to the monocalcium aluminate 10 hydrate identified earlier, several peaks for the other

calcium aluminate hydrates are also present at the end of 28 days. The peaks at 10.7 Å, 5.36 Å and 2.87 Å indicate the presence of α-dicalcium aluminate 8 hydrate while those at 4.45 Å, 3.36 Å, 2.81 Å and 2.04 Å reveal the development of tricalcium aluminate hexahydrate. The presence of tetra calcium aluminate 19 hydrate could also be noticed on X-ray trace with the peaks around 10.6 Å, 3.92 Å, 2.88 Å and 2.78 Å. Formation of calcium silicate hydrate phases is also clearly noticed. The peaks around 11.0 Å, 2.97 Å, 2.80 Å and 2.28 Å are characteristic of tobermorite while minor humps at 12.5 Å, 9.80 Å, 3.07 Å, 2.80 Å and 1.83 Å reflect the formation of C-S-H I and C-S-H II. The peaks for quartz however have not exhibited any noticeable changes in their respective intensities.

5.2.2 Black Cotton Soil from Jhansi

In addition to montmorillonite and illite, kaolinite is also present in this soil. It may be noted that the clay fraction of the soil from Banda did not have this mineral. In addition, the soil is calcareous as confirmed from the presence of the characteristic peaks for calcium carbonate at 3.04 Å, 1.91 Å and 1.88 Å and also from the effervescence observed on acid treatment.

On ageing to 7 days, the X-ray diffraction data for soil-cement samples have exhibited similar changes as in the case of Banda soil. However the amount of calcium hydroxide appears to be more in the present case,

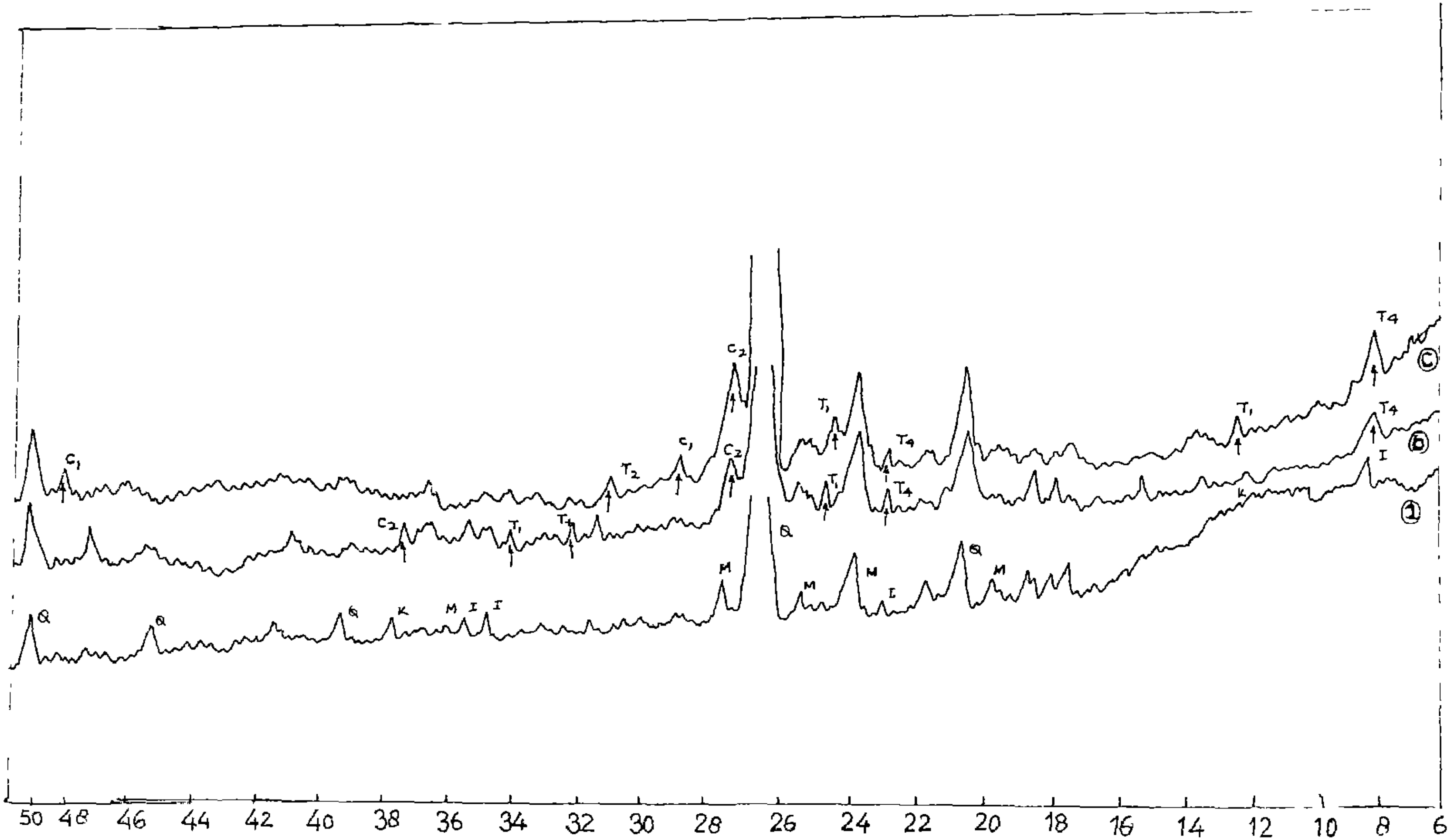


FIG. 5.2: X-RAY DIFFRACTION PATTERNS OF JHANSI SOIL SAMPLES WITH 5 % CEMENT AGED TO (a) 24 HOURS (b) 7 DAYS (C) 28 DAYS

signifying its consumption at a relatively lower rate as compared to the soil-cement samples with Banda soil. Appearance of ettringite, monocalcium aluminate 10 hydrate and α-dicalcium silicate hydrate observed in the case of Banda soil with cement, is also seen in the present case. In addition, presence of peaks at 3.92 Å, 2.88 Å and 2.78 Å reflect the development of tetracalcium aluminate 19 hydrate (Figure 5.2b).

The X-ray diffraction trace for soil-cement sample aged to 28 days is presented in Figure 5.2c. Ettringite, present in samples aged to 7 days, is not evidenced in the present case as has been the case with Banda soil with cement aged to 28 days. Monocalcium aluminate 10 hydrate is confirmed by the presence of peaks around 7.16 Å, 3.56 Å, 3.26 Å and 2.55 Å. The reflections around 10.6 Å, 3.92 Å, 2.88 Å and 2.78 Å clearly indicate the presence of tetracalcium aluminate 19 hydrate. In addition, the presence of α-dicalcium aluminate 8 hydrate is evidenced from the occurrence of peaks around 10.7 Å, 5.36 Å and 2.87 Å. Calcium silicate hydrates are also confirmed from the development of the characteristic reflections around 12.5 Å, 3.07 Å, 2.80 Å and 1.83 Å for C-S-H I and 9.80 Å, 3.07 Å, 2.80 Å and 1.83 Å for C-S-H II. It may be noticed that several of the peaks for both these compounds overlap each other and only the initial strong peak in each case occur separately.

## 5.3 CHANGES IN SOIL-CEMENT-RICE-HUSK ASH SYSTEM

Rice-husk ash has silica (quartz as the dominant phase with minor amount of tridynite) in its composition. The silica of rice-husk ash is also available for reactions with calcium hydroxide released on the hydration of cement just as in the soil-cement systems, development of new cementitious compounds is also expected in the case of soil-cement-rice-husk ash systems, on ageing to different periods of time.

### 5.3.1 Black Cotton Soil from Banda

The X-ray diffraction patterns of the Banda soil with 5 percent cement and 15 percent rice-husk ash and aged to 7, 14 and 28 days are presented in Figure 5.3. Initially the calcium hydroxide reflections are more pronounced at the end of 7 days. Broadening of illite and montmorillonite peaks on ageing and a reduction in their respective intensities are also noticed in the X-ray patterns. On ageing to 7 days, ettringite (with peaks at 5.16 Å, 3.88 Å, 2.77 Å and 2.56 Å) and monocalcium aluminate 10 hydrate (with peaks at 3.56 Å and 2.55 Å) appear to have formed in small quantities as evident from the reduced intensities of the peaks compared to the corresponding peaks in the X-ray trace for soil-cement system. The peaks around 4.22 Å, 3.92 Å, 3.54 Å, 2.87 Å and 2.80 Å indicate the formation of α-dicalcium silicate hydrate. In addition, β-dicalcium silicate appears to have developed even at the end of 7 days with

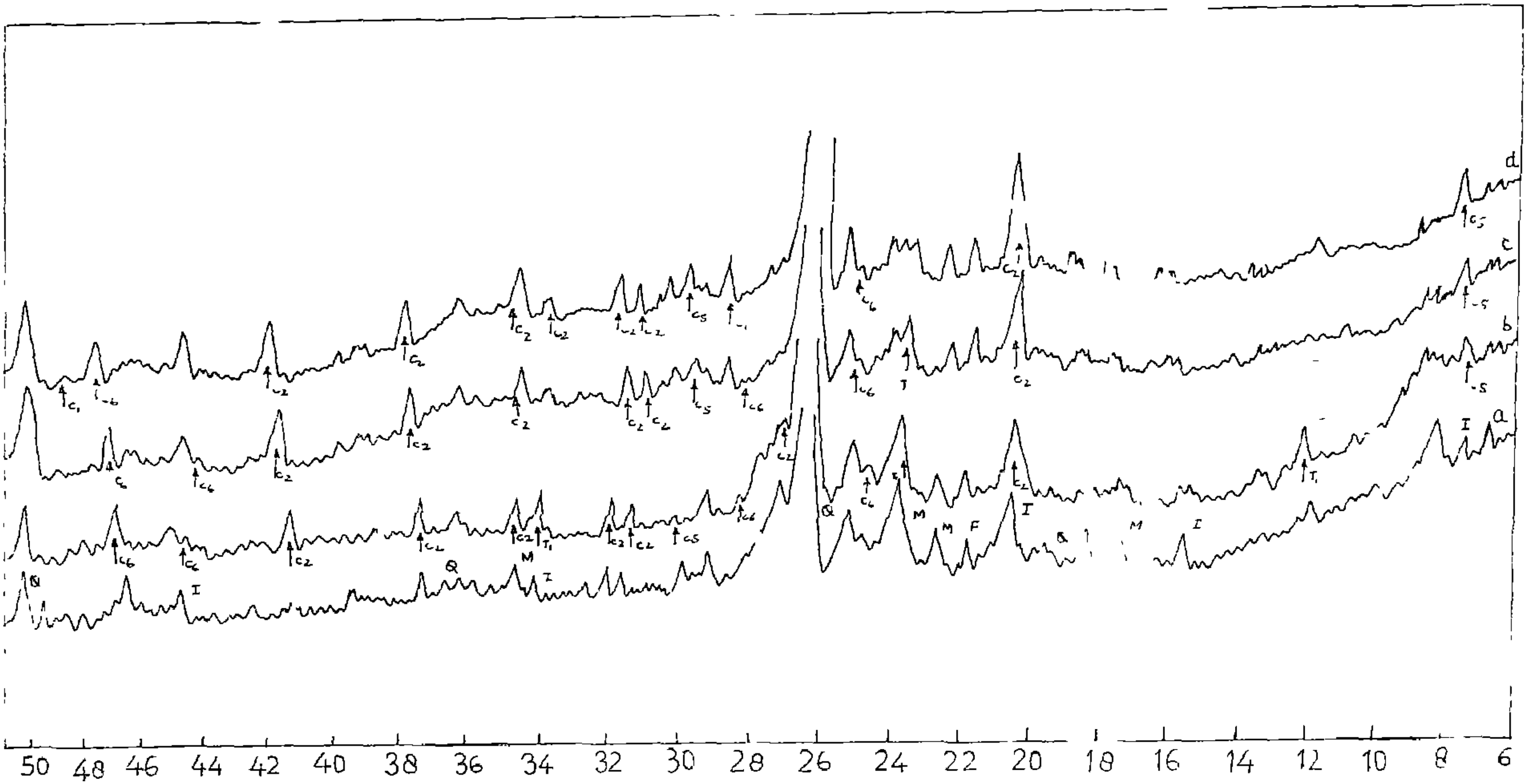


FIG. 5.3: X-RAY DIFFRACTION PATTERNS OF BANDA SOIL SAMPLES TREATED WITH 20 % OF ADDITIVES (1:3 OF CEMENT AND RICE-HUSK ASH) AGED TO (a) 24 HOURS (b) 7 DAYS (c) 14 DAYS AND (d) 28 DAYS

its reflections around 2.79 Å, 2.78 Å, 2.75 Å and 2.60 Å. In the samples aged to 7 days, presence of two more silicate hydrate phases is also clearly seen in Figure 5.3b. Xonotlite (with peaks at 3.65 Å, 3.23 Å, 3.07 Å and 2.04 Å) and tobermorite (with peaks at 11.0 Å, 2.97 Å and 2.80 Å) are also there in the X-ray trace. It may be recalled that these were not evidenced in the soil-cement samples.

On ageing to 14 days, a distinct reduction of the peaks for ettringite and monocalcium aluminate 10 hydrate is noticed (Figure 5.3c). Increase in the amount of xonotlite and β-dicalcium silicate have been evidenced from appearance of several peaks for each of these minerals as indicated below:

| | |
|---|---|
| Tobermorite | - 11.0 Å, 2.97 Å, 2.80 Å and 2.28 Å |
| Xonotlite | - 3.65 Å, 3.23 Å, 3.07 Å, 2.04 Å and 1.95 Å |
| β-dicalcium silicate | - 2.79 Å, 2.78 Å, 2.75 Å, 2.61 Å and 2.19 Å. |

Appearance of C-S-H I for the first time at the end of 14 days of ageing is clearly seen from the development of peaks around 12.5 Å, 3.07 Å and 2.80 Å.

Several distinct changes were observed in the samples aged to 28 days. The X-ray trace (Figure 5.3d) has revealed the absence of peaks for ettringite and monocalcium aluminate 10 hydrate. Xonotlite, tobermorite and β-dicalcium silicate are present in increased amount. In addition to all these products, dicalcium aluminate silicate 8 hydrate

appears to have also been formed as evidenced from presence of its characteristic peaks at 12.58 Å, 4.18 Å, 2.87 Å and 2.49 Å.

5.3.2 Black Cotton Soil from Jhansi

In the samples of Jhansi soil treated with 5 percent cement and 15 percent rice-husk ash, even at the end of 7 days of ageing very little calcium hydroxide was present in the system as evidenced from the X-ray trace (Figure 5.4b). Also the peaks of illite, montmorillonite and kaolinite exhibit broadening. Reduction in intensities of these peaks is also clearly noticeable. Reduction in amounts of ettringite and monocalcium aluminate 10 hydrate has also been observed. Development of tetracalcium aluminate 19 hydrate at the end of 7 days of ageing is also seen from presence of peaks at 10.6 Å, 3.92 Å and 2.88 Å. Some silicates have also formed with certain period of ageing. The α-dicalcium silicate hydrate, observed in the case of Banda soil treated with soil and cement aged to 7 days, has been found to be absent in the case of Jhansi soil treated with same additives. Tobermorite, xonotlite and β-dicalcium silicate hydrate are the silicate hydrates present in this case also.

The X-ray diffraction traces (Figure 5.4c) for samples aged to 14 days have revealed the reduction of intensities for peaks of ettringite and monocalcium aluminate 10 hydrate. Tobermorite, xonotlite and β-dicalcium silicate hydrate have increased in their quantity. The peaks at

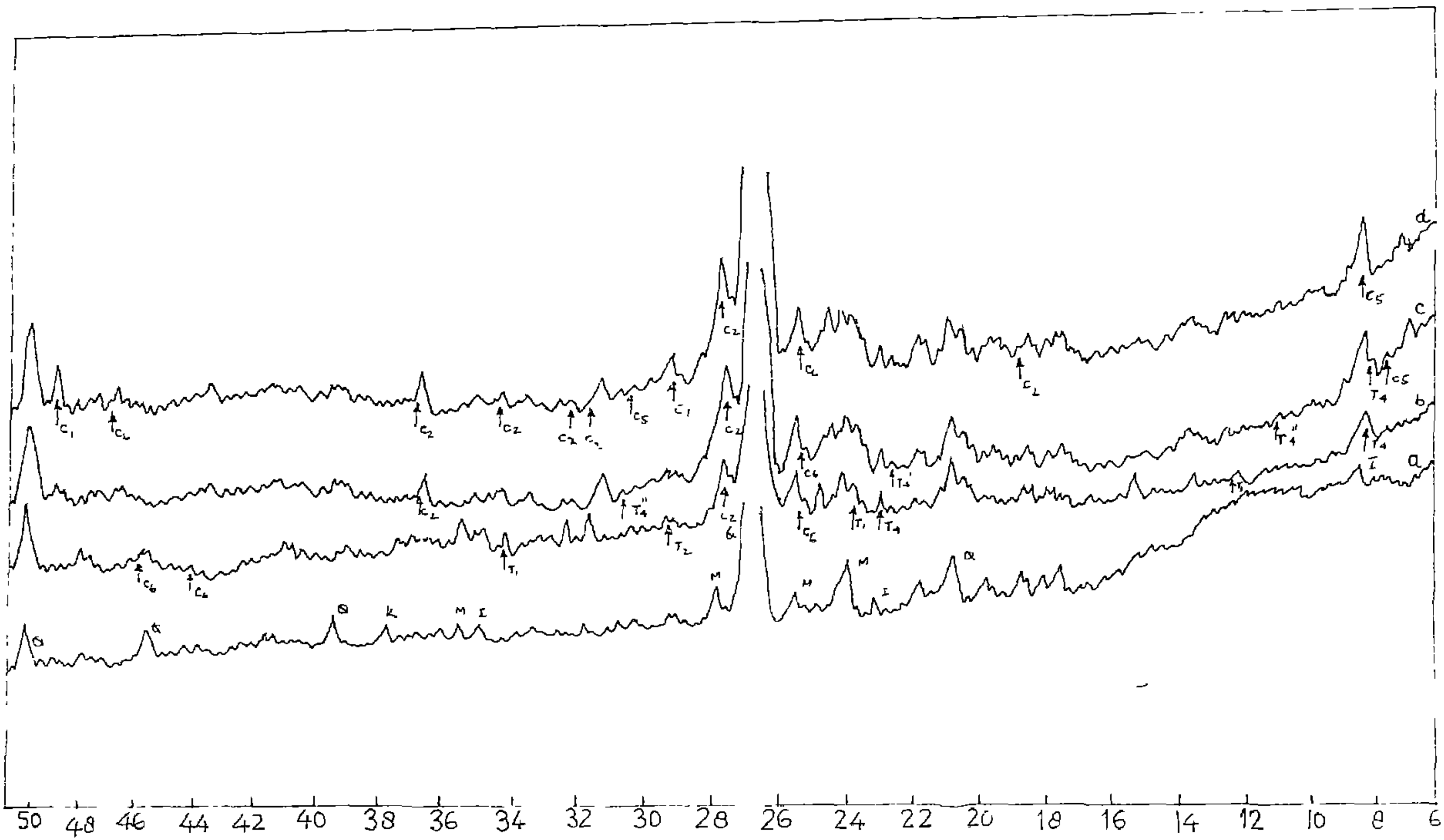


FIG.5.4: X-RAY DIFFRACTION PATTERNS OF JHANSI SOIL SAMPLES TREATED WITH 20 % OF ADDITIVES (1:3 OF CEMENT AND RICE-HUSK ASH) AGED TO (a) 24 HOURS (b) 7 DAYS (C) 14 DAYS AND (d) 28 DAYS

7.6 Å, 3.80 Å and 2.84 Å indicate possible presence of tetracalcium carbonate 12 hydrate. This product, not present in the samples with Banda soil, is perhaps due to the presence of calcite in the soil.

The changes observed in the samples aged to 28 days are similar to those in the corresponding samples with Banda soil. The peaks for ettringite and monocalcium aluminate 10 hydrate are absent (Figure 5.4d). Increase in amounts of tobermorite, xonotlite, β-dicalcium silicate, C-S-H I and C-S-H II has been evidenced from the improvement of the peaks. However the tetracalcium aluminate carbonate 12 hydrate observed in 14 days of ageing is not evidenced in present case (on 28 days of ageing) as confirmed from the absence of its characteristic peaks.

In general, the development of cementitious compounds during ageing in the soil from Jhansi on treatment with cement and rice-husk ash appears to be at a slower rate compared to the soil from Banda on similar treatment.

## 5.4 EFFECT OF CEMOS

For each of the two soils from Banda and Jhansi, specimens of soil-cement-rice-husk ash mixes have been prepared using 2 percent of cemos (stabilizer). The cement and rice-husk ash proportions have been maintained at 5 and 15 percent (by weight) respectively. Specimens have been prepared adopting same procedures as outlined earlier. Specimens aged to 7, 14 and 28 days and tested for strength have been

characterized by X-ray diffraction analysis. In each case, a set of three specimens have been tested for strength determination.

5.4.1 With Banda Soil

The X-ray diffraction trace for the sample with cement, rice-husk ash and cemos aged to 7 days is indicated in Figure 5.5b. This has clearly shown the presence of calcium hydroxide with its characteristic peaks around 4.90 Å, 2.63 Å, 1.93 Å and 1.79 Å. Ettringite and monocalcium aluminate 10 hydrate are also evidenced from the X-ray trace with their characteristic peaks. The peaks around 4.22 Å, 3.90 Å, 3.54 Å, 2.87 Å and 2.80 Å indicate the formation of α-dicalcium silicate hydrate. In addition, two other calcium silicate hydrates have also formed after 7 days. They are xonotlite (with peaks at 3.65 Å, 3.23 Å, 3.07 Å and 2.04 Å) and tobermorite (with peaks at 11.0 Å, 2.97 Å and 2.80 Å).

The specimens after 14 days of ageing exhibited reduction in intensities of ettringite and monocalcium aluminate 10 hydrate. In addition to xonotlite and tobermorite increasing in amount as indicated clearly from the increase in the intensity of their respective peaks in X-ray trace (Figure 5.5c), β-dicalcium silicate has also registered an increase on 14 days of ageing. Absence of calcium hydroxide is also quite distinct. Comparing with X-ray trace of corresponding specimens without cemos (Figure 5.3c), it is observed that the products developed are similar in both

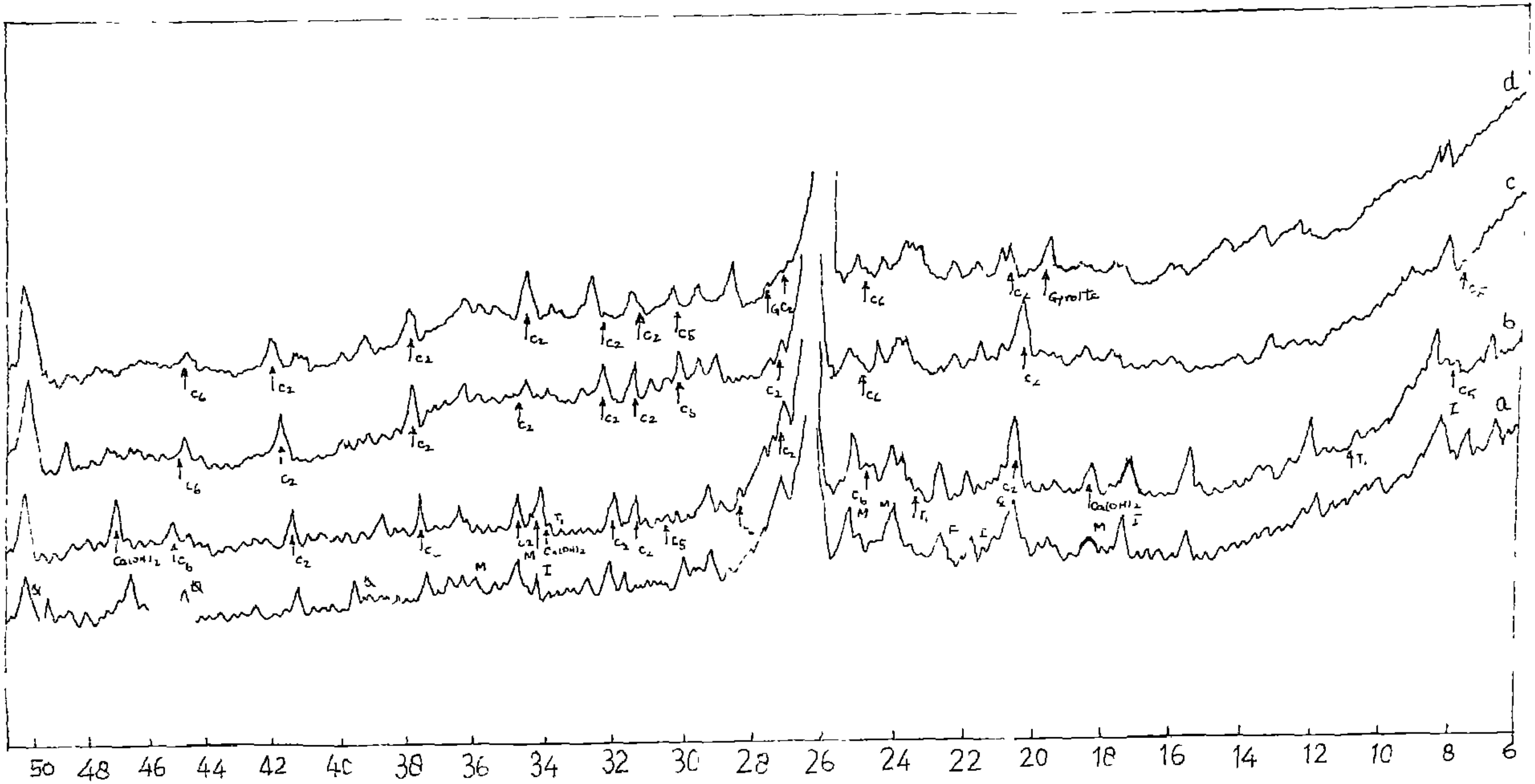


FIG 5.5: X-RAY DIFFRACTION PATTERNS OF BANDA SOIL SAMPLES TREATED WITH 22% OF ADDITIVES (2:5:15 OF CEMCS, CEMENT AND RICE HUSK ASH) AGED TO (a) 24 HOURS (b) 7 DAYS (c) 14 DAYS AND (d) 28 DAYS

cases i.e. in samples with and without cemos but the quantity of the products is slightly more in the case with cemos compared to specimens without this stabilizer.

On 28 days of ageing, ettringite and monocalcium aluminate 10 hydrate are not seen in the sample as confirmed from the absence of the characteristic peaks. Increase in the quantities of xonotlite (with peaks at 3.65 Å, 3.23 Å, 3.07 Å, 2.04 Å and 1.95 Å), tobermorite (peaks at 11.0 Å, 2.97 Å, 2.80 Å, 2.28 Å and 1.83 Å) and β-dicalcium silicate (peaks at 2.74 Å, 2.61 Å and 2.18 Å) is very well evidenced from the X-ray trace (Figure 5.5d). Gyrolite, a new product appears to have formed in the sample aged to 28 days with its characteristic peaks around 11.0 Å, 4.20 Å and 3.12 Å.

### 5.4.2 With Jhansi Soil

In the samples of Jhansi soil treated with 5 percent cement, 15 percent rice-husk ash and 2 percent cemos, at the end of 7 days of ageing considerable amount of calcium hydroxide was present in the system as evidenced from the X-ray trace (Figure 5.6b). Broadening of peaks of illite, montmorillonite and kaolinite indicate their participation in the reactions to form various new products. Ettringite and monocalcium aluminate 10 hydrate have also been observed with their characteristic peaks. Tetracalcium aluminate 19 hydrate has also developed at the end of 7 days of ageing with its peaks at 10.6 Å, 3.92 Å and 2.88 Å. Silicate hydrates are also evidenced from the X-ray trace. The

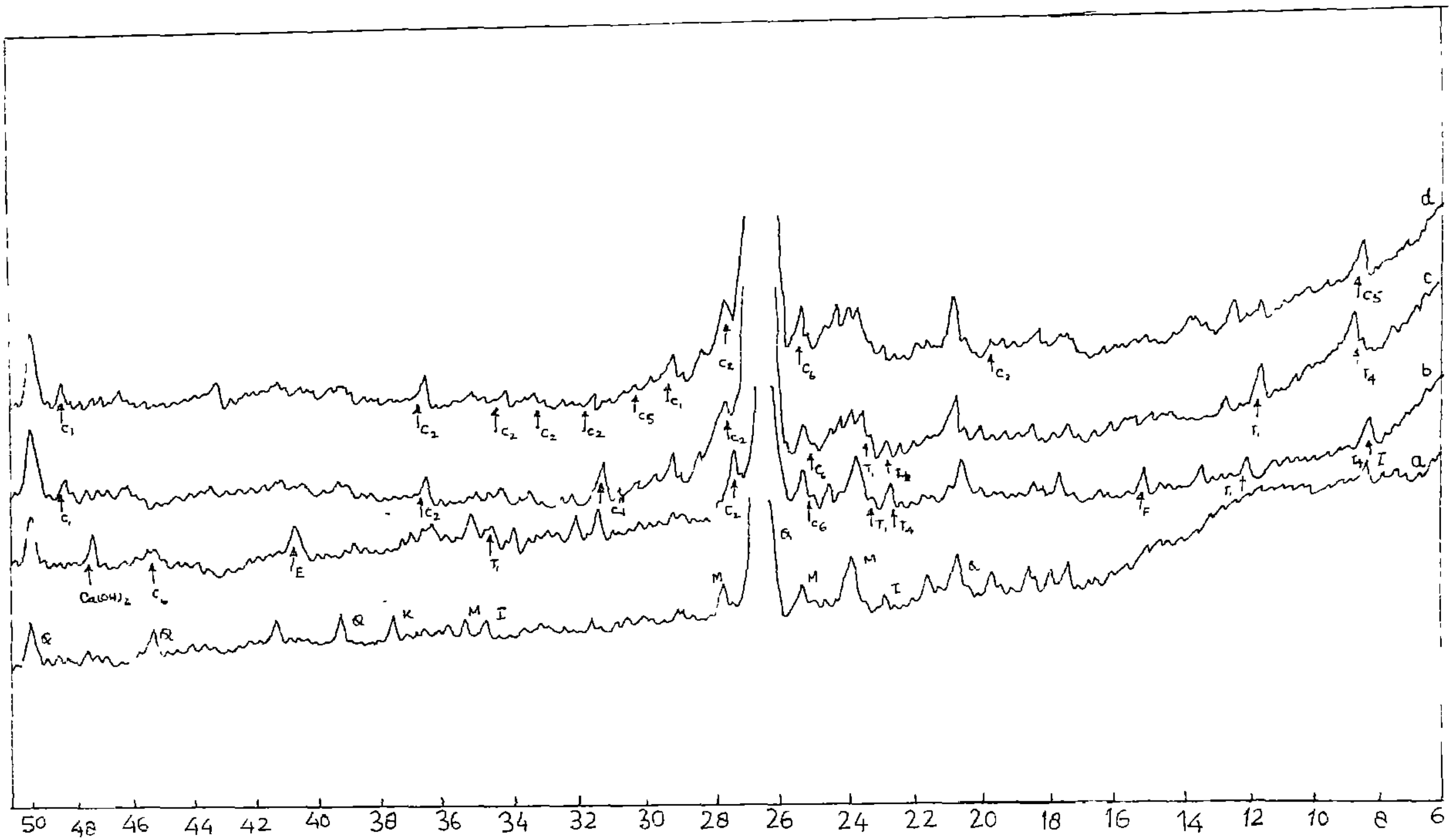


FIG 5.6: X-RAY DIFFRACTION PATTERNS OF JHANSI SOIL SAMPLES TREATED WITH 22 % OF ADDITIVES (2:5:15 OF CEMOS, CEMENT AND RICE HUSK ASH) AGED TO (a) 24 HOURS (b) 7 DAYS (C) 14 DAYS AND (d) 28 DAYS

presence of α-dicalcium silicate hydrate is not there in case of Jhansi soil whereas it was present in Banda soil. Tobermorite, xonotlite and β-dicalcium silicate hydrate are the silicate hydrates present in this case also. But as compared to Jhansi soil without cemos, the intensities of peaks of these silicates are more distinct and clear in the case of Jhansi soil with cemos.

The X-ray diffraction trace (Figure 5.6c) for sample aged to 14 days has revealed the reduction of intensities for peaks of ettringite and monocalcium aluminate 10 hydrate. Development of tobermorite (with peaks at 11.0 Å, 2.97 Å, 2.80 Å and 2.28 Å), xonotlite (peaks at 3.65 Å, 3.23 Å, 3.07 Å and 2.04 Å) and β-dicalcium silicate (peaks at 2.79 Å, 2.78 Å, 2.75 Å and 2.61 Å) are evidenced. The peaks at 7.6 Å, 3.80 Å and 2.86 Å indicate the presence of tetracalcium aluminate carbonate 12 hydrate. This product, not present in case of samples with Banda soil, is perhaps due to the presence of calcite in Jhansi soil.

The changes observed in the samples aged to 28 days are similar to those in the corresponding samples with Banda soil. The peaks of ettringite and monocalcium aluminate 10 hydrate disappear in the X-ray trace (Figure 5.6d). Tobermorite, xonotlite, β-dicalcium silicate, C-S-H I and C-S-H II with their increased intensities of peaks are evidenced on X-ray trace. Tetracalcium carbonate 12 hydrate is also evidenced to be present in the samples with cemos and it may be indicated here that this product was not

present in the sample without cemos aged to 28 days.

In general, it can be inferred that the presence of cemos in the samples has enhanced the development of cementitious compounds at a relatively rapid rate in the case of Banda soil stabilized with cement and rice-husk ash compared to the corresponding samples using Jhansi soil.

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# CHAPTER 6

# ELECTRON MICROSCOPIC STUDY

## 6.1 GENERAL

In the previous chapters, the data obtained from strength determinations and from X-ray diffraction analyses represent the changes at macroscopic and lattice levels respectively. The gap between these two stages can be effectively bridged through microscopic examination of the soil-additive system during ageing. Electron microscopy offers an excellent scope for the same.

As already mentioned the stabilization process involves reaction between the different constituents in a soil-additive system and the consequent formation of cementitious compounds during the period of ageing. The manner of reaction and the growth pattern sequence of the products in the system cannot be inferred from the X-ray diffraction data. The X-ray patterns provide information on the mineralogy of the products and the state of crystallising in addition to the quantity of material in each case. If the products are in minor quantities or in a poor state of crystallinity, difficulties in identification are encountered in the X-ray investigations. Electron microscopy provides additional information on each one of these aspects. In the present study, scanning electron microscopy has been adopted.

Electron microscopy has been increasingly used as a tool during the last two decades in soil investigations

(Croft 1964; Puseh 1966; Smart 1969; Barden and Sides 1971; Mckyes and Young 1971). Scanning electron microscopy enables the examination of intergranular pore space in addition to the grain morphology. It also enables one to understand the mode of formation of new cementitious products and the fabric interrelations between the parent and the product materials. In the present chapter, details are presented regarding the morphology of cementitious products and their manner of growth on ageing during stabilization in soil-additive systems.

## 6.2 SOIL-CEMENT SYSTEM

In the Banda soil, the clay particles are stacked in clusters and are seen as domains in the electron micrograph (Figure 6.1). The plates are slightly warped due to the expansive nature of the clay. On addition of 5 percent of cement, flocculation ensues. Even on ageing to 7 days, formation of new products is clearly seen (Figure 6.2). Development of product with needle-like structure is clearly noticeable in the central region of the micrograph. It may be recalled that the X-ray diffraction data have revealed the formation of ettringite on these samples. On further ageing, at the end of 28 days distinct changes in the system have been observed (Figure 6.3). The floc size has been considerably reduced and extensive development of the cementitious material is seen in the pore spaces. The morphology of the products appears to be different from that of ettringite in that the particles occur as hexagonal flakes at some places or as

elongated laths on other places. X-ray diffraction investigations have revealed the presence of $\alpha$-dicalcium aluminate 8 hydrate and tricalcium aluminate hexahydrate at this stage.

In the soil-cement samples with Jhansi soil, several distinct changes are noticeable. In addition to formation of ettringite, particles with partly developed hexagonal morphology are noticed (Figure 6.5) at the end of 7 days. The crystallinity of these particles improves on ageing of the sample to 28 days. Electron micrograph of this sample after 28 days of ageing (Figure 6.6) has revealed the presence of hexagonal particles (indicated by arrows in the micrograph). These particles appear to be of tetracalcium aluminate 19 hydrate. In addition to this product, development of tubular particles is also noticed. Directional growth of this product has imparted a platy structure as seen on the top right hand corner of the micrograph.

## 6.3 SOIL-CEMENT-RICE-HUSK ASH SYSTEM

Flocculation is evident in sample with Banda soil, cement and rice-husk ash at the end of 7 days. Distinct warping of the soil clay plates is also seen very clearly in Figure 6.7. On ageing to 14 days, the clay domains have a split appearence and the formation of cementitious material with fibrous structure is noticeable on the host particle surface and in the intergranular surface (indicated by arrows in Figure 6.8). X-ray diffraction investigations have revealed the formation of xonotlite. On ageing to 28 days drastic alterations within the system have been

observed. Extensive growth of tubular material (tobermorite) has taken place (Figure 6.9). The fibrous material, xonotlite, has also increased in its amount within the intergranular pore space.

With Jhansi soil, the soil-cement-rice-husk ash samples have revealed the development of particles with prismatic morphology (shown by arrows in Figure 10) and these particles appear to be α-dicalcium silicate hydrate. Development of ettringite has been clearly noticed and in addition restricted occurrence of tobermorite with tubular morphology has also been observed. On ageing to 14 days, tobermorite increases in its amount (Figure 6.11). At the end of 28 days, in addition to tobermorite, considerable growth of xonotlite is also seen as evidenced from the fibrous growth between the particles (Figure 6.12). The particle with distinct rhombohedral morphology seen in the lower right hand corner of the micrograph appears to be calcite as carbonate has also been confirmed to be present in the X-ray diffraction patterns of the soil.

## 6.4 EFFECT OF CEMOS

Presence of cemos appears to have promoted the formation of the cementitious compounds in case of Banda soil with cement and rice-husk ash in the system. The tubular material corresponding to tobermorite and considerable group of fibrous material (xonotlite) are distinctly noticeable even at the end of 7 days of ageing (Figure 6.13). The mode of

development of tubular material on the particle surface is distinctly evidenced at the end of 14 days in the micrograph (Figure 6.14). Development of the tubes and the formation of pores due to reaction are very well seen in the figure. At the end of 28 days, the system has a network of cementitious products imparting a sort of honeycomb structure (Figure 6.15). On further magnification, presence of spherical particles within the cavities (indicated by arrow in Figure 6.16) has been observed. However, presence of cemos does not appear to have accelerated the alteration and formation of new cementitious products in the case of Jhansi soil with cement and rice-husk ash as compared to similar system with Banda soil (Figures 6.17 and 6.18). A network structure is evidenced (Figure 6.19) in the case of Jhansi soil with cement, rice-husk ash and cemos on ageing to 28 days.

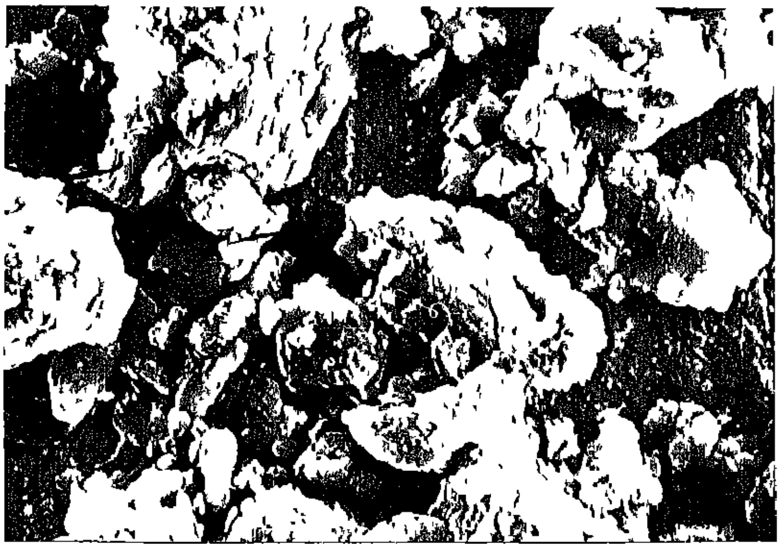

Fig. 6.1. Clusters and domains of clay particles in Banda Soil (SEM, X1500).

Fig. 6.2. Development of reaction product with needle-like structure in Banda soil with 5% cement aged to 7 days (SEM, X3000).

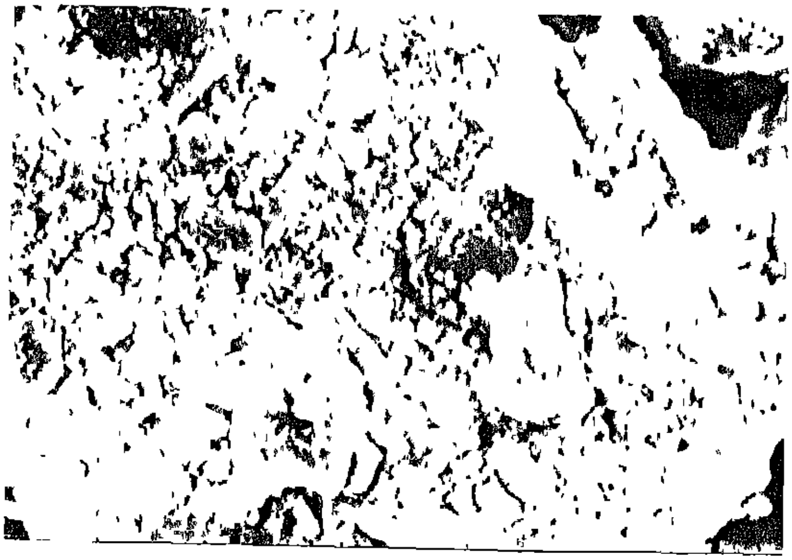

Fig. 6.3. Reduced flocs in Banda soil with 5% cement aged to 28 days (SEM, X1500).

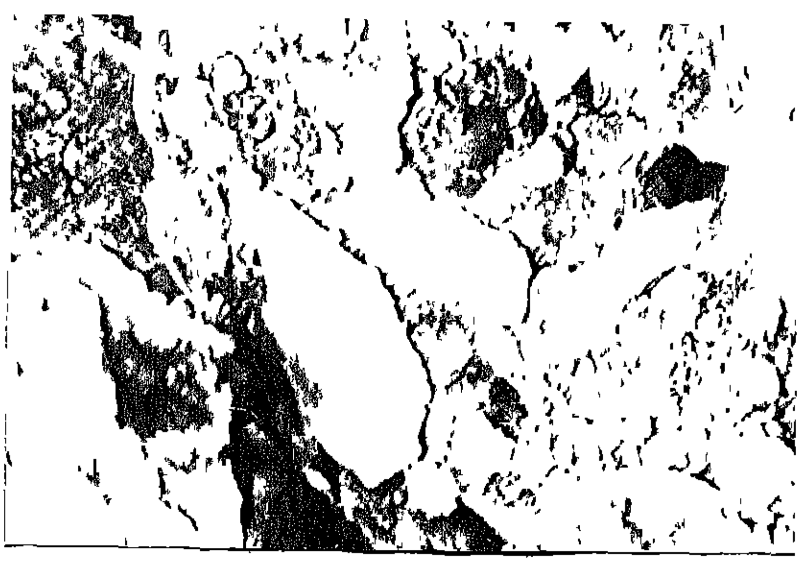

Fig. 6.4. Clay particles in Jhansi soil (SEM, X2000).

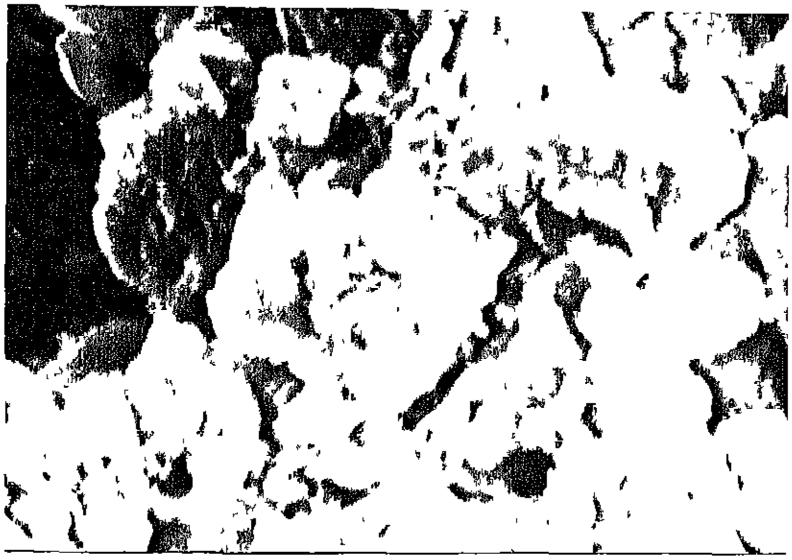

Fig. 6.5. Particles with hexagonal morphology in Jhansi soil with 5% cement aged to 7 days (SEM, X1500).

Fig. 6.6. Directional growth of hexagonal particles in Jhansi soil with 5% cement aged to 28 days (SEM, X1500).

Fig. 6.7. Warping of clay particles in Banda soil with 5% cement and 15% rice-husk ash aged to 7 days (SEM, X3000).

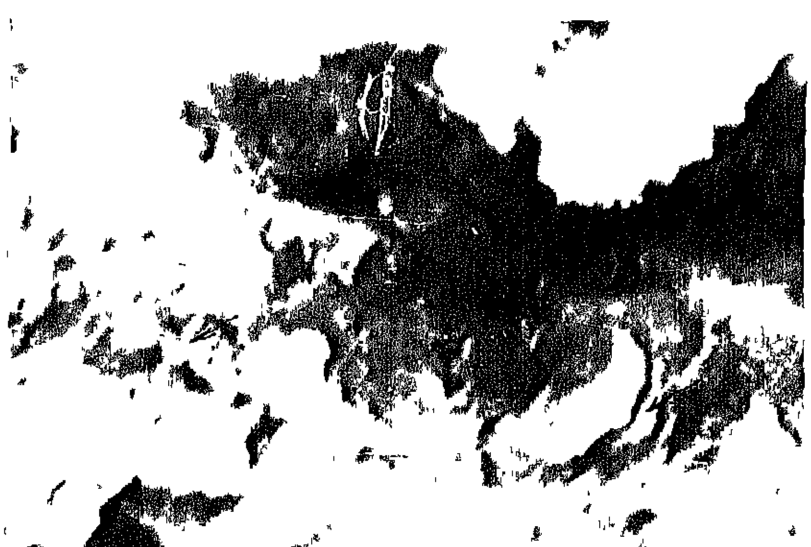

Fig. 6.8. Clay domains in Banda soil with 5% cement and 15% rice-husk ash aged to 14 days, with the formation of fibrous product (SEM, X1500).

Fig. 6.9. Growth of tubular material in Banda soil with 5% cement and 15% rice-husk ash aged to 28 days (SEM, X1500).

Fig. 6.10. Growth of cementitious products in Jhansi soil with 5% cement and 15% rice-husk ash aged to 7 days (SEM, X1000).

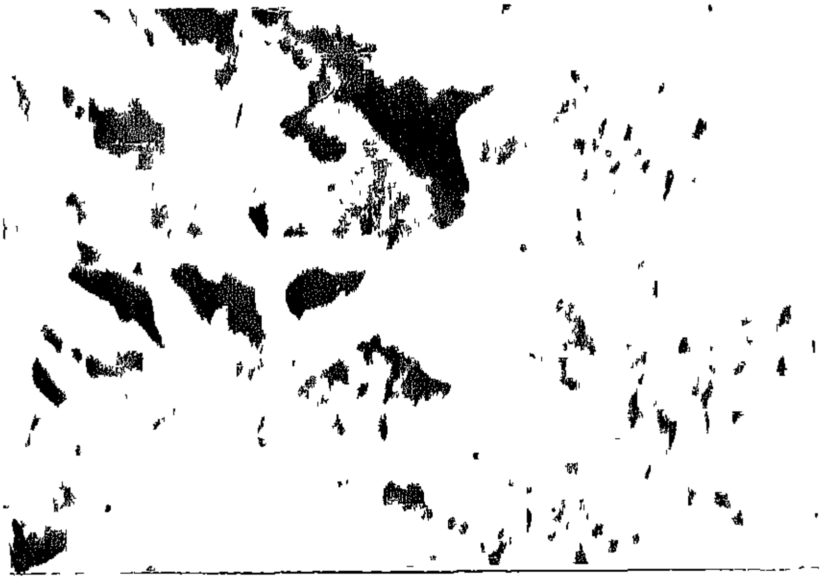

Fig. 6.11. Tubular growth in Jhansi soil with 5% cement and 15% rice-husk ash aged to 14 days (SEM, X1000).

Fig. 6.12. Reaction products with tubular and fibrous morphology in Jhansi soil with 5% cement and 15% rice-husk ash aged to 28 days (SEM, X2000).

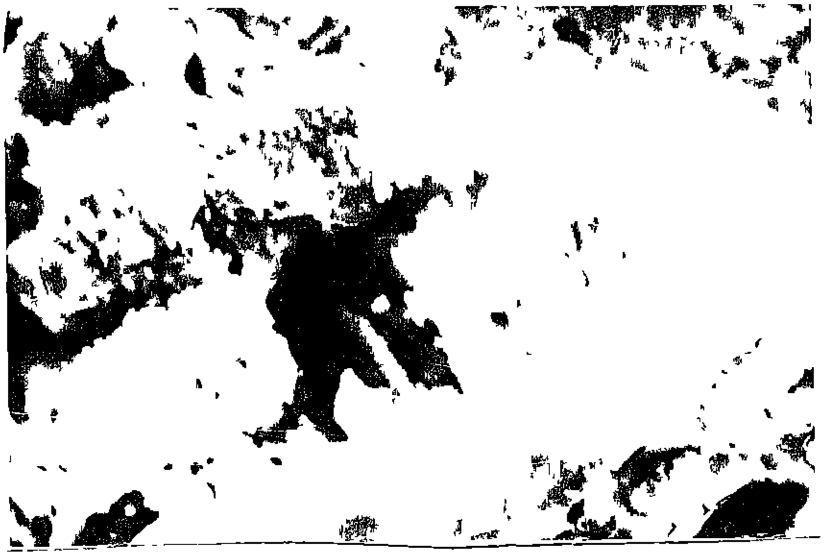

Fig. 6.13. Growth of tobermorite with tubular morphology in Banda soil with 5% cement, 15% rice-husk ash and 2% cemos aged to 7 days (SEM, X1500)

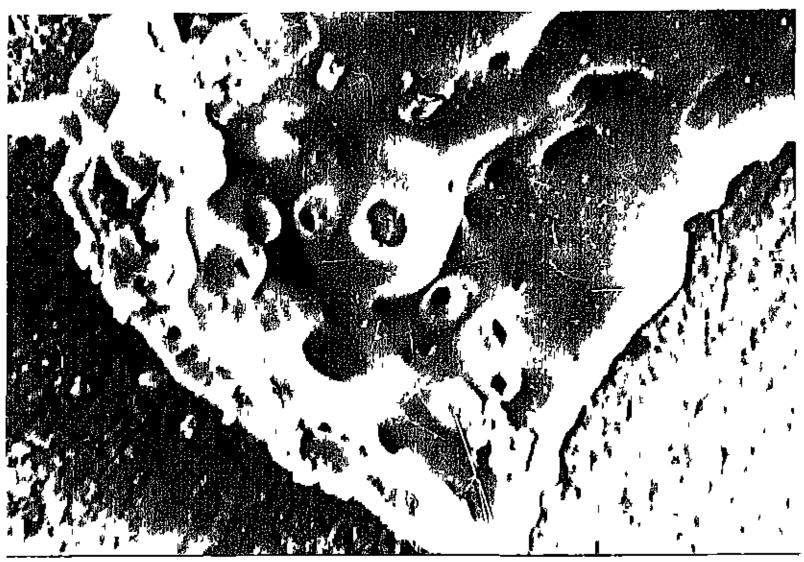

Fig. 6.14. Reaction pattern in case of Banda soil with 15% rice-husk ash, 5% cement and 2% cemos aged to 14 days (SEM, X2500).

Fig. 6.15. Honeycomb structure in Banda soil with 5% cement, 15% rice-husk ash and 2% cemos aged to 28 days (SEM, X1500).

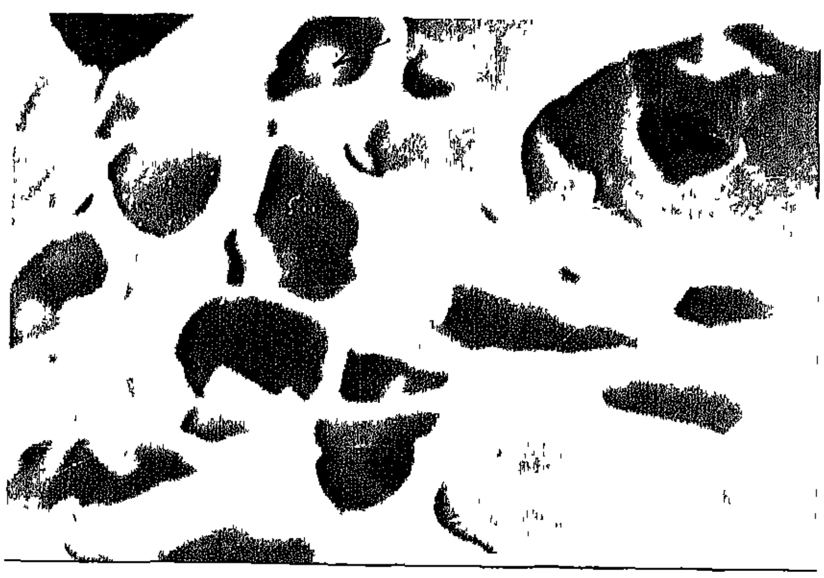

Fig. 6.16. Enlarged version of the previous figure indicating presence of spherical particle in the cavity (SEM, X4500).

Fig. 6.17. Reaction products in Jhansi soil with 5% cement, 15% rice-husk ash and 2% cemos aged to 7 days (SEM, X3000).

Fig. 6.18. Formation of products with tubular morphology in Jhansi soil with 5% cement, 15% rice-husk ash and 2% cemos aged to 14 days (SEM, X1500).

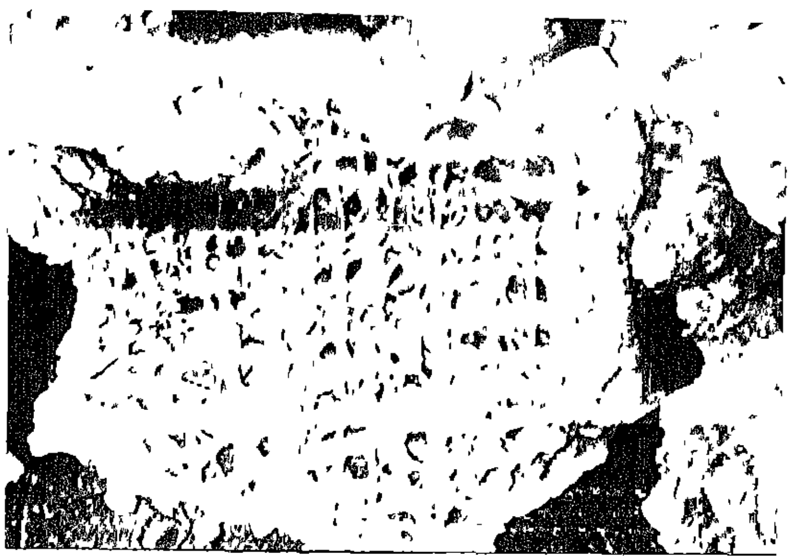

Fig. 6.19. Skeletal structure formed in Jhansi soil with 5% cement, 15% rice-husk ash and 2% cemos aged to 28 days (SEM, X1500).

# CHAPTER 7

## INTEGRATED PICTURE

The soils used in the present study are from Banda and Jhansi in U.P. Both of them are of low strength and expansive in character. Montmorillonite is the dominant clay constituent occurring along with illite. In addition, kaolinite in minor quantity is present in the soil from Jhansi. In the dry state, these two soils have very low unconfined compressive strength values (around 0.725 $Kg/cm^2$ for Jhansi soil and 0.600 $Kg/cm^2$ for Banda soil). Addition of cement and rice-husk ash during stabilization results in several changes -- both of short term and long term in nature within the soil-additive system.

Portland-Pozzolana cement is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite with small amount of gypsum (as evidenced from X-ray diffraction pattern). Hydrated silicates and hydrated aluminates of different kinds form as reaction products in cement hydration. Lime released on hydration of cement during stabilization process enhances the pH of the soil-additive system and also induces flocculation of the clay constituents. This has been confirmed from the scanning electron micrographs.

The soil-cement, soil-cement-rice-husk ash and soil-cement-rice-husk ash-cemos systems are thus alkaline and contain free calcium hydroxide. The increase in pH of the

systems facilitates greater solubility of silica and alumina. Under the alkaline conditions, the silica of the soil clay and the silica of rice-husk ash have greater solubility.

The soil-cement, soil-cement-rice-husk ash and soil-cement-rice-husk ash-cemos stabilization processes involve the cement-soil clay interactions. Hydrated cement-soil mixtures differ from the concrete in that the aggregate in the latter case is largely inert and the strength of the concrete is solely due to the hydration products of the cement and their bonding properties with the aggregates. The pH in the hydrated cement thus remains at a constant level thoughout the curing. The situation is different in the case of soil-cement stabilization. The soil-clay-cement interactions are largely governed by the pH of the system. The reactions are thus pH dependent. As the cementitious products form in the clay-cement interactions, the pH of the system continues to decline. This is the case in both the soils taken up for stabilization in the present study.

The X-ray diffraction patterns of both the systems indicated broadening of the clay mineral peaks and a decrease in their respective intensities on ageing reflecting their participation in the reactions. The X-ray diffraction patterns also revealed the formation of cementitious compounds during ageing. Different calcium aluminate hydrate and calcium silicate hydrate phases are formed in addition to tobermorite depending on the type of soil constituents and the additives. The quantities of the respective compounds are observed to increase with time of ageing. In the initial stages of reaction, peaks for compounds are very broad indicating their 'gel'

like nature and their crystallinity improves with time. Thus in the present study, it has been established that the cementitious products are contributed both by hydrated cement as also by cement-clay and cement-rice-husk ash interactions.

Among the reaction products in the soil-cement system, ettringite and monocalcium aluminate 10 hydrate are observed as the dominant ones in both the soils. However, as the reactions proceed on ageing, ettringite disappears due to its metastable nature. Such observations has also been reported by Grudomo (1964). On ageing to 28 days, formation of α-dicalcium aluminate 8 hydrate, tetracalcium aluminate 19 hydrate, C-S-H I and C-S-H II has been evidenced in soil-cement system in case of both the soils. Soil-cement combination has revealed an increase in strength with ageing upto 28 days. The rate of strength gain in both the soils is correlated with the rate of formation of the cementitious products. The soil from Banda registers maximum strength value of around 15 $Kg/cm^2$ with 5 percent cement on ageing to 28 days while the same is only around 9 $Kg/cm^2$ in the case of Jhansi soil. The relatively rapid increase in strength in the first 14 days can be attributed to the flocculation and rate of reaction. The reaction rate appears to be slower after 14 days.

Addition of rice-husk ash to the soil-cement system brings about marked changes. Presence of reactive silica and alumina in the ash facilitates greater solubility. A faster pace of reaction could be confirmed from the rapid consumption of calcium hydroxide as also the drastic alteration of clay minerals as evidenced in the X-ray diffraction traces.

The quantity of the cementitious products is also more in each of the cases with cement and rice-husk ash together as additives. While C-S-H I and tobermorite have appeared at a later stage in the case of soil-cement systems, tobermorite develops much earlier (within the first 7 days) in the case of soil-cement-rice-husk ash system. Formation of xonotlite is a distinct feature in presence of rice-husk ash within the system. Increase of strength has also been evidenced with cement and rice-husk ash as additives during stabilization. On ageing to 28 days, soils from Banda and Jhansi have exhibited strength values of 19.2 and 11 $Kg/cm^2$ respectively with these additives.

Distinct changes have been observed when cemos is used as a stabilizer in the soil-cement-rice-husk ash system for both the soils. Acceleration of reactions has been evidenced from the drastic changes in the intensities of reflections in X-ray pattern of the starting materials signifying the increased consumption of the materials. Increase in amount of the reaction products was also evidenced. Tobermorite has developed even at the end of 7 days. In addition to bermorite, xonotlite and $\beta$-dicalcium silicate, gyrolite is a new compound formed after 28 days of ageing in case of Banda soil. This soil has gained the strength value around 23.2 $Kg/cm^2$ with cement, rice-husk ash and cemos after 28 days of ageing whereas Jhansi soil could reach upto 13.2 $Kg/cm^2$ after the same period, clearly indicating the pronounced effect of cemos in case of Banda soil with cement and rice-husk ash as additives.

Scanning electron microscopy has enabled an understanding of the changes in the soil-additive system during

stabilization. Distinct changes in the fabric with increasing reaction could be seen. On ageing to 28 days, the fabric attains a honeycomb type in the case of soil with cement and rice-husk ash and cemos as additives. Development of reaction products has also been clearly evidenced from the distinct morphological characteristics of the reaction products. Ettringite appears with needle-like structure while tubular morphology is typical of tobermorite. Distinct hexagonal flakes of tetracalcium aluminate 19 hydrate and the fibrous structure of xonotlite are clearly seen in the scanning micrographs. The growth pattern of the reaction products has also been clearly observed. The product develops on the surface of the particles as well as in the intergranular pore space.

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